nlgi spokesman

Some Aspects of Bentone Greases

By R. E. FARISS, Baroid Division, National Lead

Technical Considerations in the Marketing of Rear Axle Lubricants

By C. M. HEINEN, Chrysler Corporation

Traffic Safety Through Enforcement

By RAY ASHWORTH, Traffic Institute, Northwestern University

1957 NLGI Committees

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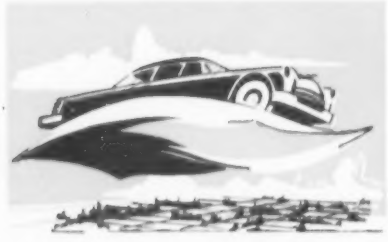

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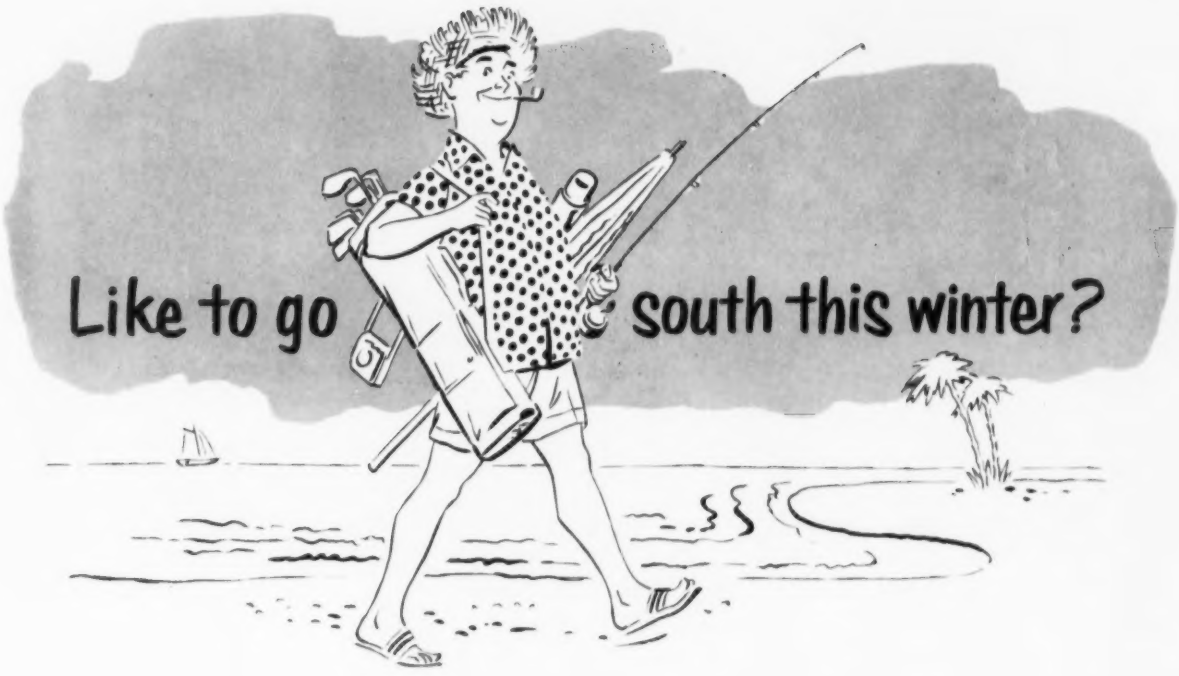

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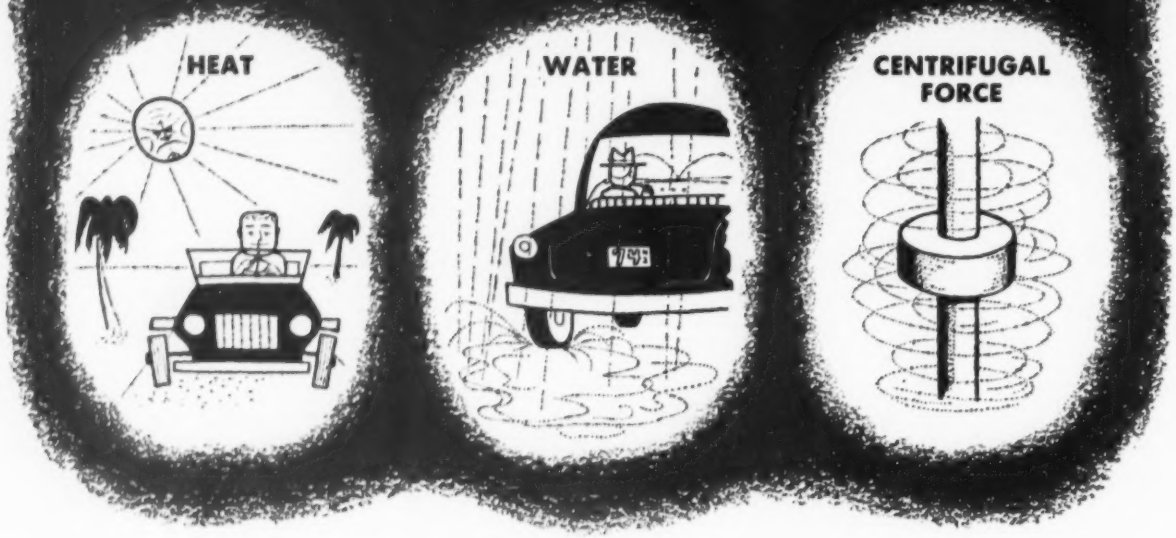

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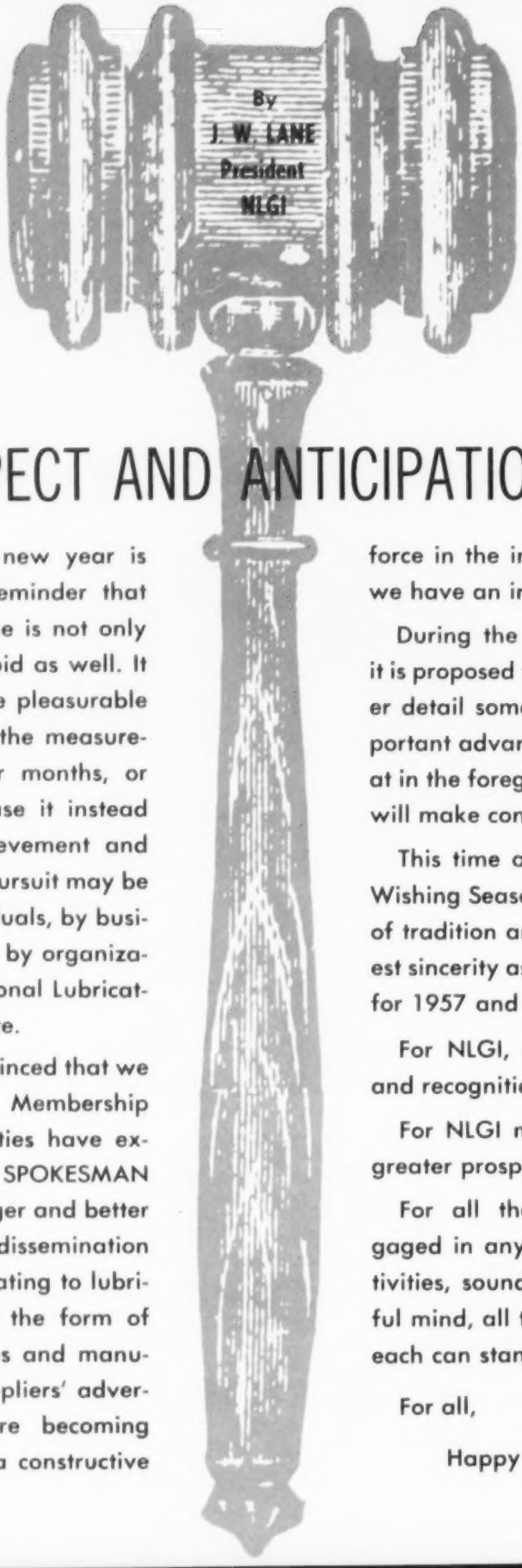


RETROSPECT AND ANTICIPATION

The start of a new year is often a painful reminder that the passing of time is not only inevitable, but rapid as well. It can be much more pleasurable I think, to forego the measurement in years, or months, or weeks, and to base it instead on terms of achievement and satisfaction. This pursuit may be enjoyed by individuals, by business and industry, by organizations such as National Lubricating Grease Institute.

I am firmly convinced that we are progressing. Membership has grown, activities have expanded, the NLGI SPOKESMAN is becoming a bigger and better medium for the dissemination of information relating to lubricating greases in the form of contributed articles and manufacturers' and suppliers' advertisements. We are becoming better known as a constructive force in the industries in which we have an interest.

During the next few months it is proposed to discuss in greater detail some of the more important advances merely hinted at in the foregoing. I predict this will make convincing reading.

This time of year is also the Wishing Season. So, in the spirit of tradition and with the greatest sincerity as well, I wish these for 1957 and beyond:

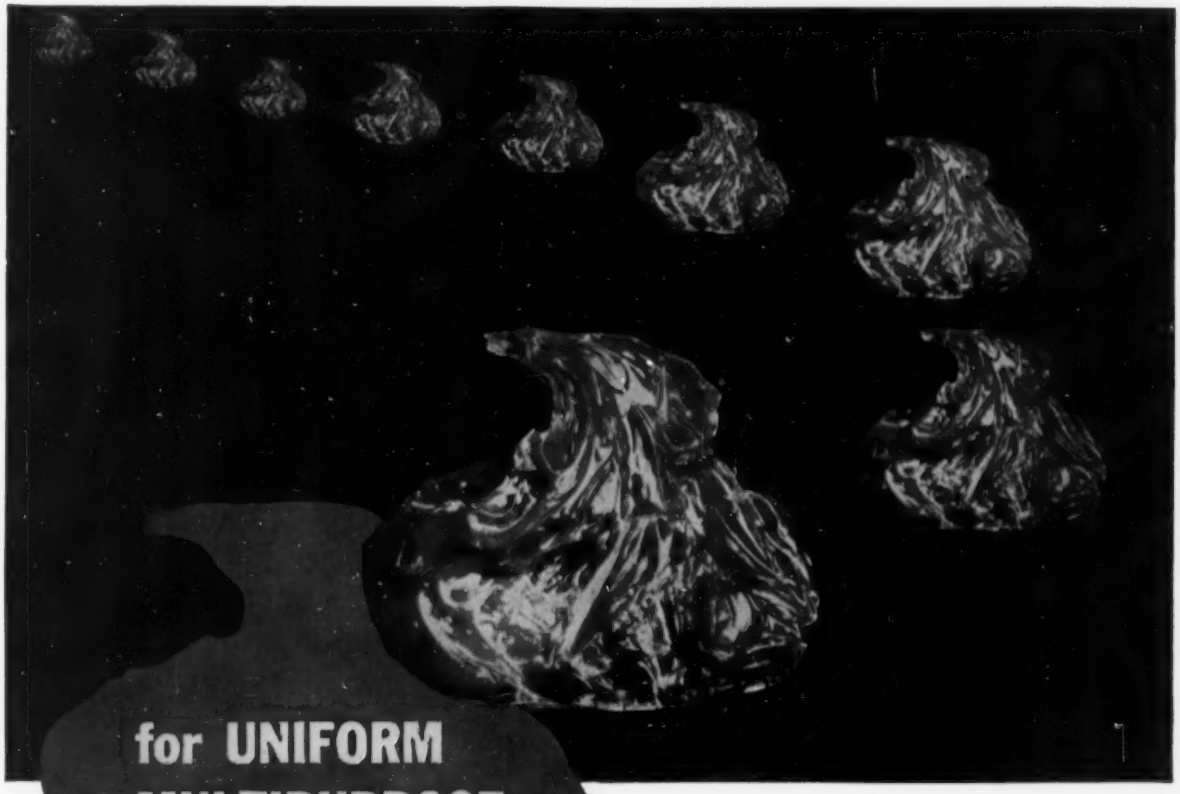
For NLGI, continued growth and recognition;

For NLGI members, an even greater prosperity;

For all the individuals engaged in any way in NLGI activities, sound health, a peaceful mind, all the prosperity that each can stand; and,

For all,

Happy New Year!



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THE COVER

NLGI's newest masthead is introduced this month with a scene familiar to the industry . . . a sample of the product. The new design itself is another in a series of editorial improvements enacted through the years for a better magazine and is patterned after the Institute's popular new letterhead. We feel it incorporates many new and modern ideas to give the NLGI SPOKESMAN distinctive quality and strength. A new feature is the listing of technical titles on the front cover to grant easier reference, filing and accessability for our readers.

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Bentone

Introduction

THE USE OF organophilic bentonites (known commercially as "Bentones") in the preparation of lubricating greases was presented before the National Lubricating Grease Institute in a paper[1] by McCarthy and Finlayson. A more detailed discussion of the nature of these materials was presented in a series of papers[2,3] entitled "Organophilic Bentonites" by Jordan et al. Compondu[4] has described the use and advantages of a commercial Bentone grease.

Bentone is a registered trademark of the National Lead Company, applied to a class of products resulting from cation exchange reactions between organic bases and bentonite or its clay mineral component, montmorillonite, the individual products being identified by a number following the mark "Bentone." The description of these materials and how to prepare them has been given by Hauser[5] and their application for bodying organic media has been described by Jordan[6]. Important properties of gelled systems containing Bentone, such as absence of melting point, lack of phase changes at any temperature, resistance to water and stability of gel structure on standing or under mechanical working, have been presented[1,7].

In the formation of these products an exchange reaction occurs between sodium bentonite and an organic ammonium or onium salt to yield an organic onium bentonite plus salt. Insolubility of the onium bentonite compound favors the reaction to the right. The most versatile material is *Bentone* 34, which is dimethyldioctadecyl ammonium bentonite. It will gel many organic liquid systems such as aromatic hydrocarbons and various aliphatic liquids such as petroleum oils. To prepare a Bentone grease it is necessary to disperse the Bentone 34 throughout the oil system, either by mechanical or chemical means. The following discussion will treat principally with some of the factors involved in gelling with Bentone 34 those petroleum oils used for lubrication purposes.

Milling

Differential Mill. The earlier experimental greases were prepared by mixing Bentone 34 and oil and passing the mixture over a differential three roll paint mill. The original slurry contained approximately 33⅓% Bentone 34 and produced a very heavy paste by this method. This concentrate was then diluted to the desired final concentration and remilled until homogeneous. Although excellent greases were prepared from very small amounts of Bentone 34 by this method the production rate was so low as to make the method not too attractive economically.

Colloid Mill. A number of colloid mills, jet homogenizers and related milling equipment, some of which had already found application in the milling of soap base greases, were then tried. The greases described in the McCarthy-Finlayson paper were prepared by passing a mixture of oil and Bentone 34 through a colloid mill until a grease was produced. This method produced satisfactory greases but required more Bentone

About the Author

One of the chief developers on Bentone greases is Robert E. Fariss, of the Baroid Division of National Lead Company. Fariss has been a member of the firm since 1951. He had previously worked in the production research department of the Texas Company. Fariss received a B. S. in chemical engineering from Rice Institute in 1942 and worked as a research chemical engineer for Dow Chemical Company until 1944. He joined the Naval Reserve and served as navigator and communications officer until 1946, when he re-entered Rice and was awarded a Ph. D. degree in 1950.

Greases

By R. E. FARISS
Baroid Division, National Lead

34 than the method utilizing the three roll mill. In some instances incomplete dispersion was achieved in the mill and the greases continued to body in application.

Recent Trends. The more recent procedure is one in which a dispersant is added to the Bentone 34-oil mixture to chemically aid in dispersing the Bentone. This is then followed by milling as in the above procedure. Care must be used in the selection and use of these dispersants in order to obtain maximum yield from the Bentone 34 used. These dispersants have been found to be, in general, low molecular weight polar organic compounds. Depending upon the choice of oil, dispersant and the care in preparation, excellent greases can be prepared in this manner with a better yield than by the simple mixing technique. A comparison of three methods is shown in figure 1.

For the purpose of this study a 40% by weight blend of a solvent refined neutral (gravity: 30.1° API @ 60° F,viscosity at 100° F: 343.7 SUS, viscosity index: 94) and a solvent refined bright stock (gravity: 22.4, viscosity at 100° F: 3280, viscosity index: 71) was prepared. The resultant blend had a gravity of 26.1, viscosity at 100° F, 1065, V.I. 78. The greases prepared by the three roll differential mill method were milled twice at 33⅓ per cent Bentone 34 and twice at the

Figure 1

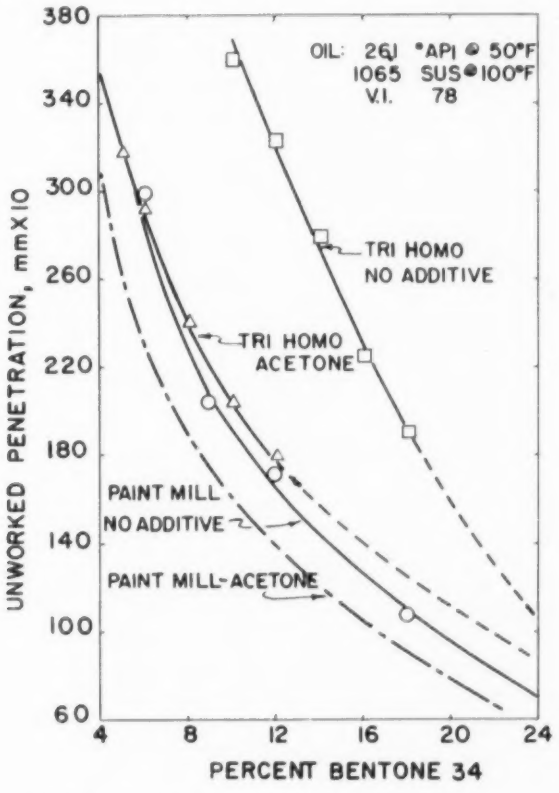
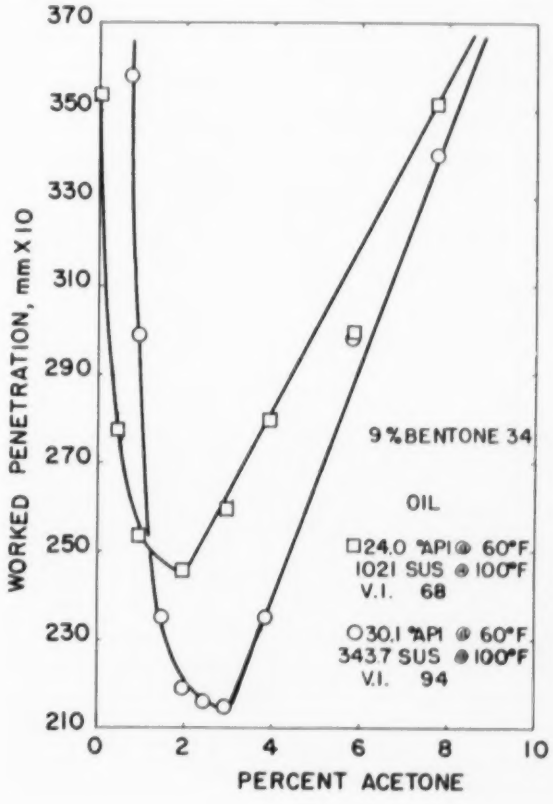


Figure 2



desired concentration. For comparison the results using acetone as a dispersion aid in the premix by the same method are shown as a dotted line in figure 1. The samples prepared by the colloid mill technique (with and without dispersant) were mixed 30 minutes and then milled at 0.4 lb. per min. through a No. 2 Tri Homo Disperser set at 0.001 inch rotor-stator clearance. All samples were allowed to remain overnight prior to measurement. From these data the efficiency of the process relative to the differential mill procedure with dispersant was calculated and is given in table 1.

Effect of Base Oil

Refining. Mention has been made that care should be exercised in the selection and use of the dispersant. Depending upon the extent and the nature of the refining process, the ease of dispersion and the amount of dispersant required are found to vary widely with the base oil. This is shown graphically in figure 2. It is evident that the more highly refined oil requires more additive but is apparently less sensitive to the amount required in the region of optimum gelation.

The relationship between the worked and unworked penetrations is shown in figure 3. Since essentially the results are the same regardless of which values are used it was decided to use the 60 stroke or ASTM worked penetration in presenting these data.

Figure 3

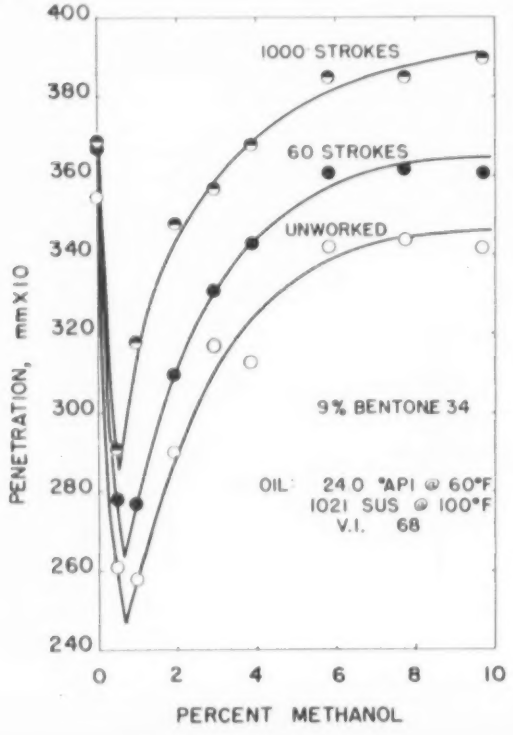
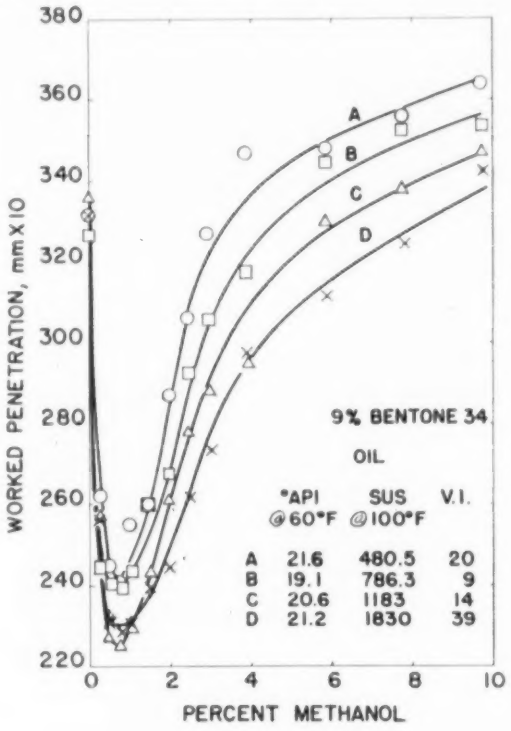


Figure 4



Viscosity. In addition to the refining and nature of the base oil some variation in yield is noticed when base oils of different viscosities are used under the same conditions of preparation. This is shown in figure 4 where four oils produced in the same manner and from reportedly the same crude are compared. Comparison of the curves shown in figure 4 indicates that a minor gain in yield is to be expected with an increase in viscosity. This may be attributed in some degree to the increased shear forces achieved in the colloid mill by the more viscous material, the increase in shear producing an increased degree of dispersion. It is of interest to note that the optimum yield is a function of the

per cent methanol only and not of the oil viscosity.

In the evaluation of a number of oils a record was kept of the per cent Bentone 34 required to prepare a grease having an unworked ASTM penetration of 300. These are shown in figure 5. Approximately 70% of the samples tested were with ± 0.5% of the average shown as the heavy line in figure 5.

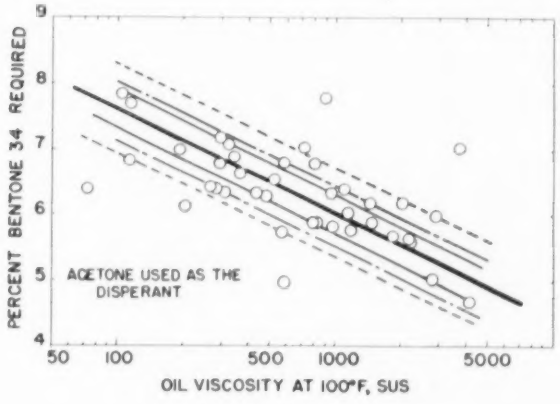
Under conditions of maximum dispersion the consistency of the produced Bentone 34 grease is essentially independent of the viscosity of the base oil provided the oils are relatively similar in composition. This is shown in figure 6.

These samples were prepared by the technique previously discussed using the differential mill and a dispersion aid. This demonstrates that under conditions of maximum dispersion the consistencies of the greases produced from a 1021 SUS coastal stock oil and from a 3283 SUS solvent refined bright stock are the same.

Dispersion Aids

The foregoing discussion has pointed out the advantages of utilizing mills capable of extreme attrition to

Figure 5



achieve the best and most economical greases from Bentone 34. In order to obtain a reasonable production rate it is necessary to utilize less efficient milling technique. It has been shown how this could be overcome to some extent by the use of a dispersion aid. These compounds have been passed over briefly but any discussion relative to the production of Bentone 34 greases would be incomplete without a more detailed presentation of the advantages and disadvantages of dispersion aids.

Description. As previously mentioned the dispersion aids are in general low molecular weight, polar organic compounds. In the previous illustrations acetone and methanol, two of the more commonly used dispersants, were chosen. Both compounds are low boil-

Figure 6

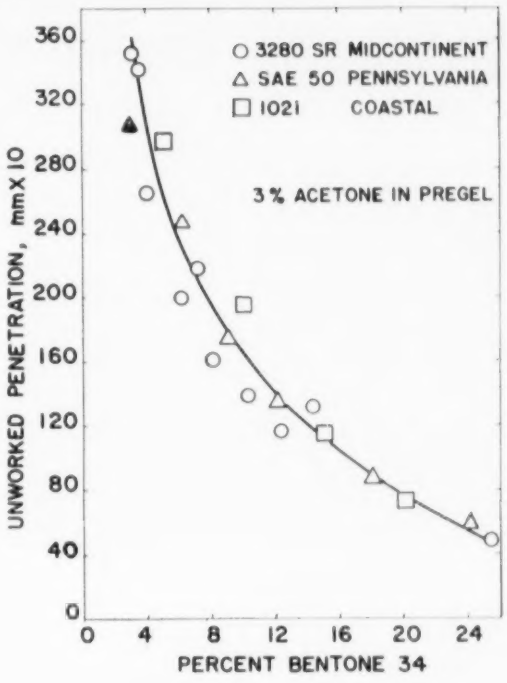
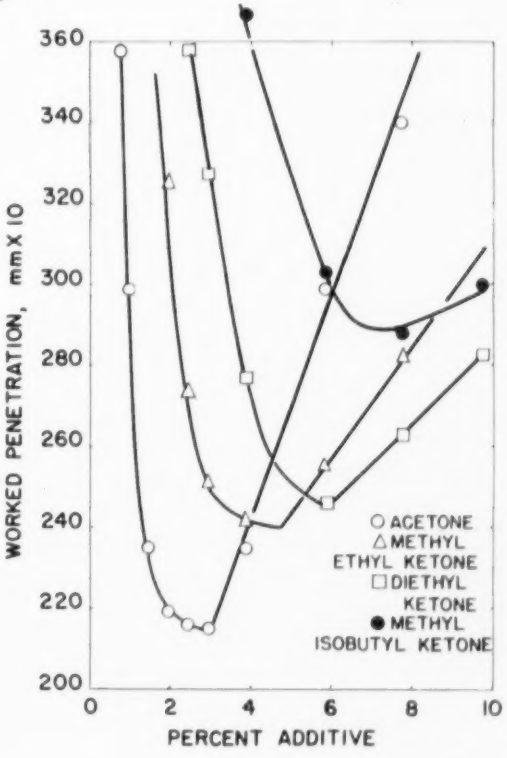


Figure 7



ing and require reasonable precautions in handling. For a given homologous series the efficiency of the dispersion aid apparently decreases with increasing molecular weight. This has been treated in detail by Jordan [2,3] but the treatment was restricted to systems other than the Bentone 34-lubricating oil type.

The effect of molecular weight is shown in figure 7 for the system Bentone 34-oil. It is clearly shown that as the molecular weight is increased either the efficiency of the dispersant diminishes or the requirements become excessive.

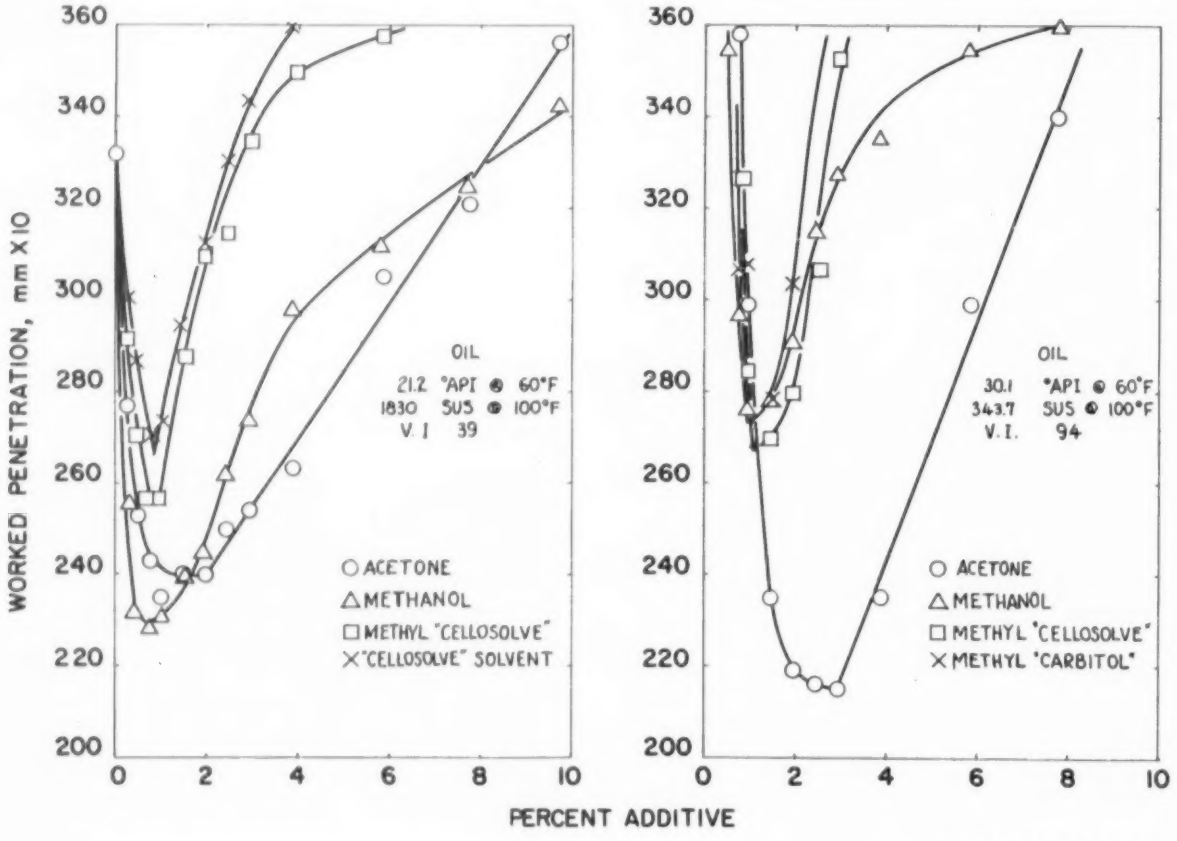
Effect of Oil Stocks. To demonstrate the different behavior of the dispersion aids in oil stocks of varied physical properties a solvent refined neutral and a conventionally refined coastal oil were chosen. These oils are shown in figure 8.

Here again the influence of the oil is noted. The conventionally refined coastal oil gives optimum yield with methanol whereas the solvent-refined neutral gives an appreciably better yield with acetone. In both oils acetone gives a satisfactory yield as contrasted to the marked decrease in the effectiveness of methanol in the solvent refined neutral. It is also evident that more additive is necessary for optimum results in the high gravity oil than in the low gravity oil.

Effect of Polarity and Molecular Weight. The ether alcohols shown in the above series present an interesting comparison with methanol. This series demonstrates that the amount required for optimum gelation is essentially independent of the molecular weight but that the efficiency decreases with increasing molecular weight. The relationship obtained with members of the ketone family is shown in figure 9.

For this study the solvent refined neutral oil was chosen. Acetonylacetone (hexanedione 2, 5) and acetylacetone (pentanedione 2, 4) present an interesting case. Both of these compounds are diketones. For optimum gelation approximately the same amount of pentanedione 2, 4 as acetone is needed whereas less hexanedione 2, 5 is required. Further, the hexanedione 2, 5 is more efficient than the pentanedione 2, 4 although the hexanedione 2, 5 has a greater molecular weight.

Figure 8



It may be of interest that this compound has a boiling point of 378° F.

Effect of Bentone 34 Concentration. The dependence of the dispersion aid upon the concentration of the Bentone 34 is shown in figure 10. It is seen from figure 10 that the ratio of dispersant to Bentone 34 is not constant, the amount of dispersant at the optimum changing only slightly for an increase of Bentone 34 from 6 to 10%. The amount required is apparently related to the polarity of the base oil although the successful polar compounds to date have been water soluble compounds.

Preparation of Premix. In addition to the milling procedures outlined in the previous sections there are many variations possible for a given procedure which may greatly influence the yield obtained for a given system. A brief discussion of these methods will be given. The four best known dispersion aids were chosen and evaluated using different techniques with a colloid mill. The results of these tests are given in table 2.

These samples were prepared using the solvent refined neutral oil previously described: Method 1 con-

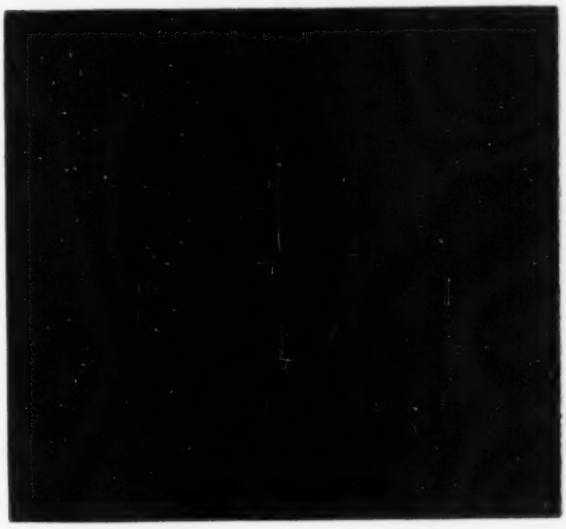


Figure 9

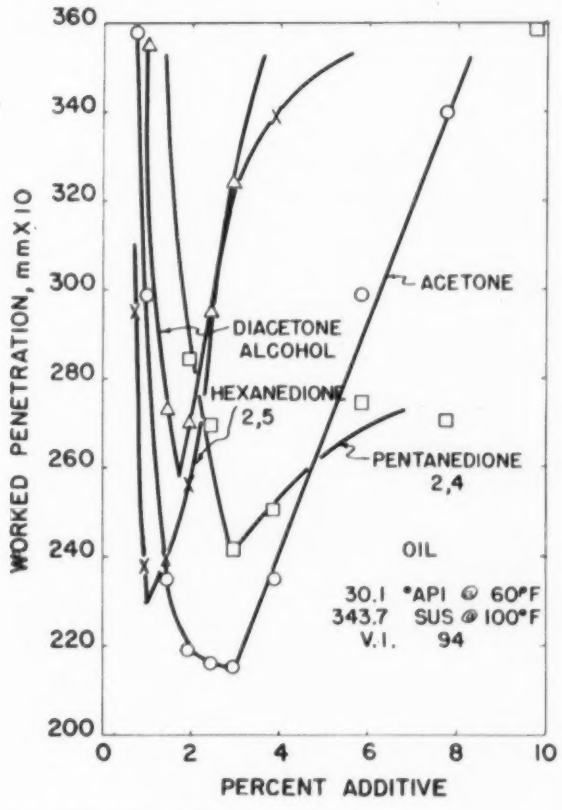
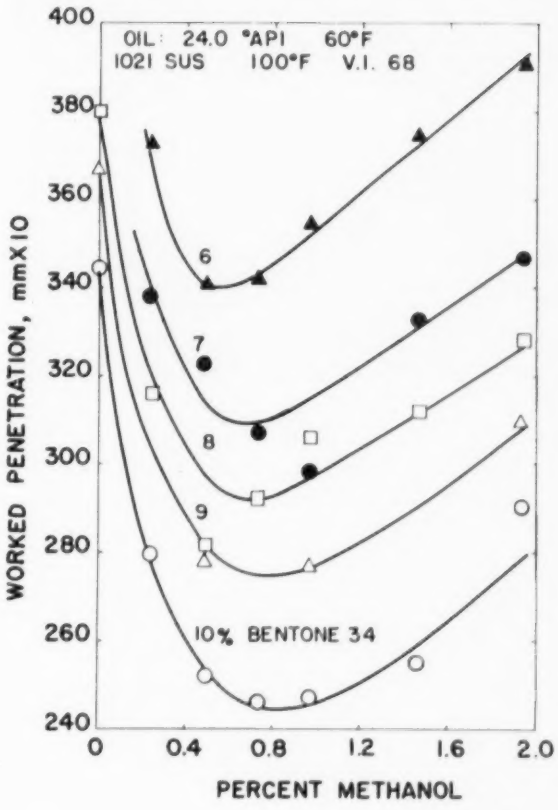


Figure 10

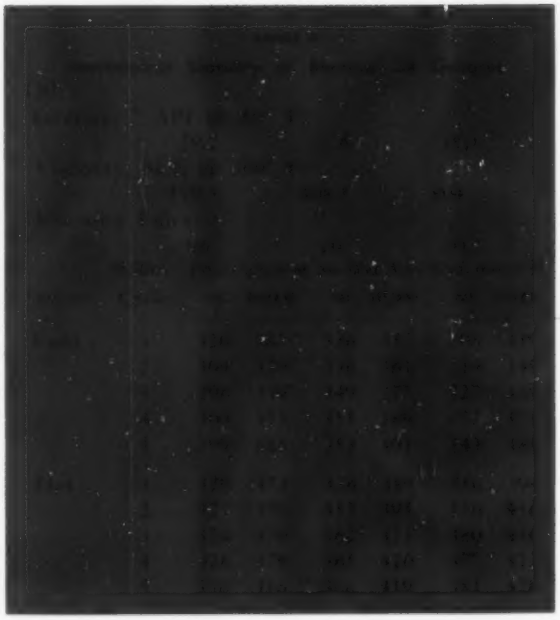


sisted of slurrying the Bentone 34 and oil, adding the dispersant and stirring for 30 minutes prior to milling. Method 2 consisted of dispersing the polar compound in all the oil and then adding the Bentone. For method 3 the Bentone was slurried with ⅓ the oil, the dispersant added, and stirred until a heavy paste was formed. The remaining oil was then added. After 30 minutes stirring the mix was milled as in the previous methods. With the exception of methanol the best results were obtained by method 3. For this system the less mechanically stable greases were obtained with methanol as the dispersion aid.

Effect of Heat. Another variation possible is heating to remove any volatiles present. This is sometimes used in conjunction with method 3. The success of this method depends largely upon the base oil. In general, highly refined oils give excellent results whereas, poorly refined oils have been found to give very erratic results. It is usually advantageous to add 0.1% water on cooling prior to milling. The amount and the temperature at which addition of the water should be made depend on the system used. The results obtained by this procedure are shown in table 3.

These data indicate that the blend used consisting of 33% solvent refined neutral (viscosity: 343.7 SUS @ 100° F) and 67% solvent refined bright stock, (viscosity: 2383 SUS @ 100° F) gave the better yield when 0.04 to 0.09% water was used as specified above.

Mechanical Stability. The results obtained with sample 5 proved of interest in that they indicated that the breakdown as determined by the ASTM Method D-217-52T was not permanent but represented an orientation of some type. To check this samples were prepared in three different oils, worked and remilled through 5 cycles. Samples were prepared both by the cold process and the heated process described above. These data are shown in table 4.



The penetration measurements were made immediately upon completion of the working specified. These data indicate that under conditions of turbulent shear action the breakdown of a Bentone 34 grease is negligible. For the highly refined oil sample 3% acetone was used; 1.5% was used for the others.

Summary

The production of a Bentone 34 grease is thus related to the method of preparation, the oil used, the type of mill employed and whether or not dispersants are employed. Once these relationships have been established the production of greases with Bentone 34 is a simple and routine process easily controlled. If other additives, such as anti-oxidants, corrosion inhibitors, EP's, tackiness agents, etc., are to be added best results are obtained by using special procedures for their incorporation.

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TECHNICAL CONSIDERATIONS IN THE MARKETING OF REAR AXLE LUBRICANTS

By C. M. HEINEN, Chrysler Corporation

Presented at the Ninth Annual IOCA Meeting in Chicago

TRANSPORTATION IN DAYS of old was not very complicated. A bale of low-octane hay, an occasional shot of water, and the knights were off in pursuit of loot, fair maidens or—if they needed a good excuse to get out of the house—dragons. In spite of this, our good friend Shakespeare tells us that a battle was lost because a horse dropped a nail, and the party line to Ye Olde Service Station was tied up by one fair damsel telling another what she heard about that terrible knight in the third castle down the road—or something.

But the general problems of transportation in this modern era are far more complex—and, therefore, all the more thought provoking. I would like to consider what I believe to be one of the most interesting areas of mutual problems which we have at present—rear axle lubrication.

Why do I believe it so? Certainly not because of its economic importance. As you can see from Figure I, it is a pygmy financially, when compared to motor oils or fuels and about even with Type A fluids which are also being discussed today. Because they are so complex? I think not. Many other fluids are harder to make.

Probably the reason gear oils are so interesting to me at present is that after a long period of comparative dormancy, everything about them is being questioned simultaneously. I might add that some very illuminating answers are developing.

FIGURE I

Approximate 1955 Usage of Petroleum Products

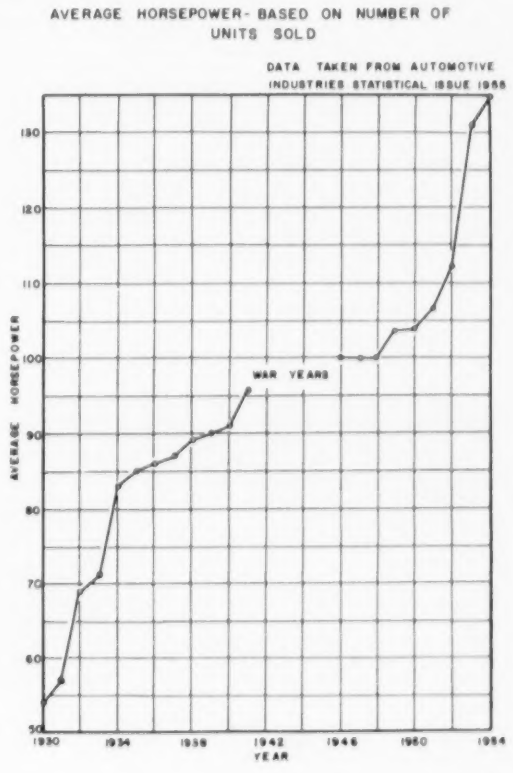
Motor Fuel, All	54,329,744,000 gal.
Motor Oils, All	880,624,000 gal.
Gear Oils	49,200,000 gal.
Type A Fluid	25,747,000 gal.
TOTAL OIL	955,571,000 gal.

These data were obtained from various sources.

Let us start out by looking at the fundamentals which have caused this ferment in the thinking about rear axle lubricants.

1. Increased output of modern engines has caused greater power to be put through the rear axle. Figure II shows that this increase has been very steep.
2. Car weights are going up slowly, but steadily, as

Figure 2



can be seen from Figure III. This means greater inertia loads.

3. Car heights are going down. This is shown in Figure IV. This in turn means greater offset and smaller rather than larger differentials.
4. Rear axle temperatures are rising. This is confirmation of the fact that the other trends are affecting the work being done through the rear axle. The temperature rise is shown in Figure V.
5. Better highways are available on which higher average speeds are possible. These probably result in longer periods of high temperature operations, although this is difficult to prove statistically.

When these facts are considered without regard to service consequences they are of importance to the technician only. When they coincide with a record of service failures, they assume more than academic interest. This is what happened about five years ago. The result has been a re-evaluation of test techniques, lubricant formulation, and lubrication theory which promises to alter completely a situation which had been unchanged for about ten years. But let's not get ahead of the story.

Shortly after World War II, U. S. Army Specification 2-105B was issued. It described a universal gear lubricant suitable for Army purposes. The specification was based primarily on performance type tests. Included among others were tests for copper activity, foaming, channeling, compatability, and two very important full scale axle tests.

The two latter tests were almost identical with the Coordinating Research Council's L-19 and L-20 procedures. To refresh your memory the L-19 consisted of a series of acceleration–deceleration cycles at 40-10 m.p.h. and 80-60 m.p.h. using a Chevrolet vehicle and axle combination. This test was intended to evaluate the load carrying characteristics of an oil under high speed operating conditions. The L-20 test was intended to evaluate oils under considerations of high torque and low speeds. It was based on the use of a three-quarter ton Dodge axle operating for twenty-four hours under about 32,000 inch/lbs. of torque at 62 r.p.m. and at 200° F. This test was intended for use on an axle dynamometer and operated on the drive side of the gears for twenty-four hours.

In theory at least the two tests covered the opposing requirements which a universal gear labricant must meet. At high speeds, sudden shocks occur which raise the local temperatures to the melting point of the metal. The lubricating film is ruptured, and the chemical activity of the lubricant must be such that a reaction at the high spot has to take place instantaneously or welding of the metal surfaces will occur. If this does not happen a condition known as scoring will take place. A "scored" gear is shown in Figure VI.

Figure 3

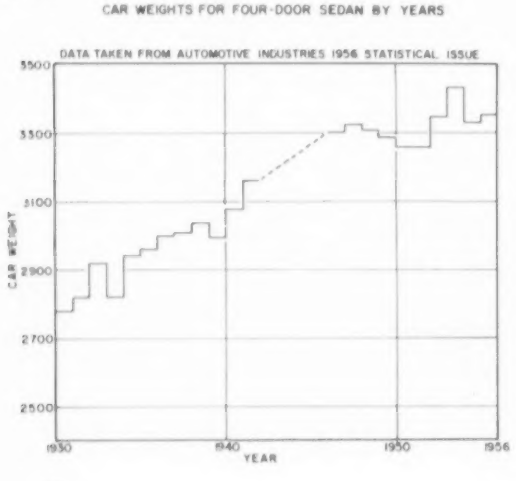


Figure 4

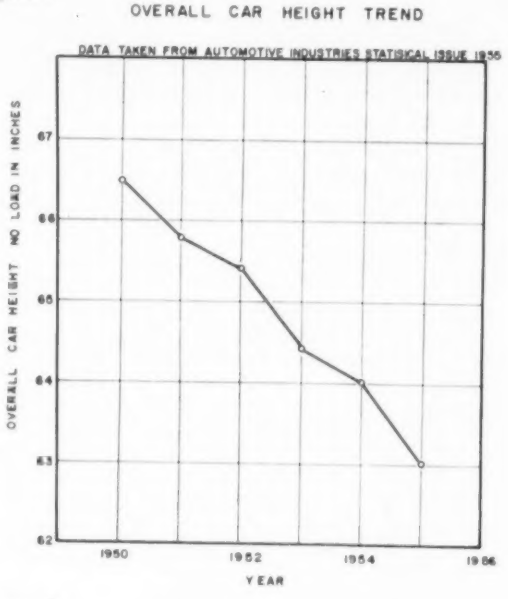
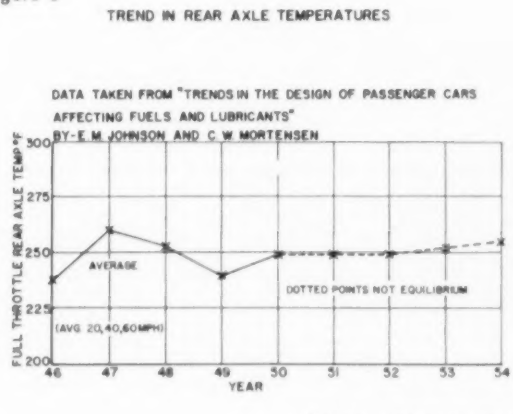


Figure 5



You will note that the score mark is a localized phenomenon.

It would appear that the solution to this type of failure would be a lubricant of greater chemical activity at high temperatures, and as a matter of fact, it is. Unfortunately, however, most lubricants that will react more rapidly at extremely high local temperatures will also react more rapidly at lower temperatures. This brings about other problems. The L-20 is intended to evaluate the conditions under which this lower surface temperature activity is apt to cause trouble.

Chemical reactions produce a film involving both the gear lubricant and the gear metal. As the film reaches a certain thickness, the shear forces become strong enough to break part of it off. High activity lubricants will then react further to produce more film and eventually a substantial loss of metal occurs. This is demonstrated qualitatively by noting the metal loss which occurred when comparing the action of a high activity lubricant of the active sulfur type with several universal gear lubricants on a Falex machine wear test. Figure VII shows the results.

In actual practice truck operation is often of such a type that high torques are combined with high bulk oil temperatures and low speeds. Wear of the type shown in Figure VIII results. It should be noted that an extreme pressure film is also required for this type of operation, but that due to the low speeds involved, it can be put on at a more moderate rate, consequently a less active lubricant can be used.

The problem has two possible solutions:

1. A lubricant which would have sufficiently rapid activity to take care of the high temperature condition and whose reaction rate would decrease sufficiently so as not to cause excessive reaction at lower temperatures.

FALEX MACHINE WEAR TEST

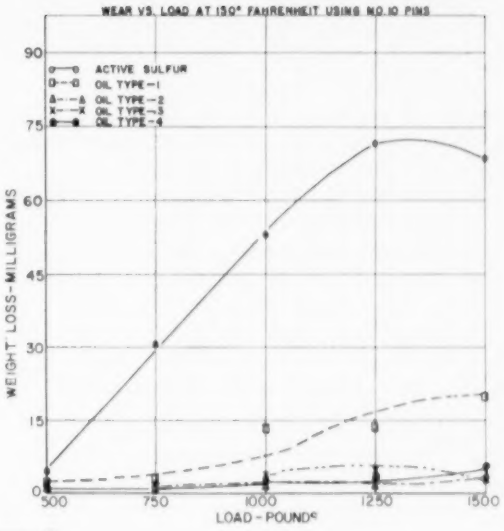


Figure 7

2. Lubricants of different performance levels for truck and passenger car differentials.

Until 1951 the lubricants defined by the military specification appeared to meet both requirements satisfactorily. This quality level became the generally accepted service station oil and was sold under the name of "multi-purpose." Discussions of gear problems were directed primarily at three areas:

1. The reproducibility of the L-19 test and the correlation of the L-19 test as run on the dynamometer and in the field.

2. The significance of the corrosion tests and the field evidence to justify them.

Figure 6—Scored Teeth

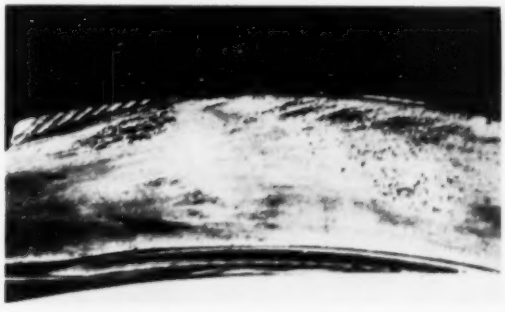


Figure 8—Severe Wear Sample

Photos courtesy of Lubrizol Corporation

3. Gear oil stability as affected by ambient and axle temperatures.

In 1951-52 several events occurred which indicated that the oils described by the military specification were not satisfactory in all applications and that modifications in test procedures and oil formulation were required. These included:

1. An Army report of "scuff" type failures in trucks operating at 50-60 m.p.h. Lest this appear too surprising, let me remind you that in terms of pinion r.p.m. this is equivalent to a substantially higher car speed. Figure IX shows this relationship.
2. A confidential survey by an oil company revealed that axle failures on the automobile industries' new models were rising.
3. It was announced that the axle being used for the L-19 test was to be withdrawn from the market and another would have to be substituted.
4. Considerable evidence of oil deposits as a result of high temperature was beginning to accumulate.

It became apparent that universal gear oils did not have enough chemical activity to supply the necessary chemical films under the new peak torque conditions. Several solutions were tried to supplement the film. These included:

1. Operation for a short period of time with active sulfur lubricant followed by a drain and universal gear oil fill.
2. Forming a phosphate film on the gears during production and using universal gear oil. This was our own solution.
3. Recommending active sulfur lubricant for the life of the car.

It is the third recommendation which causes the most trouble from a merchandising point of view. As has been indicated before, an active sulfur type lubricant is not satisfactory for truck operation, and since many of the trucks on the road come to service stations for lubrication, two lubricants would have to be carried.

Two types of lubricants result in greater expense and, what is even worse, considerable confusion. As a test case I asked one of my associates to go to six stations of one of the major oil companies and get me some truck type gear lubricant. He tried six and came back with three phosphorus-chlorine oils, one an SAE 140, an SCL type, and active sulfur type and a straight mineral oil.

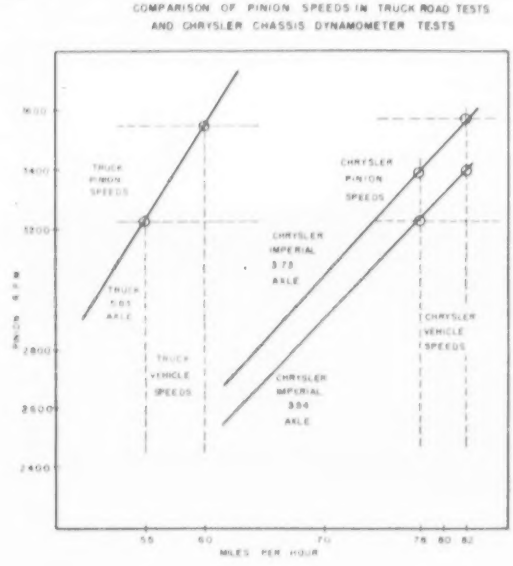
These indications of probable confusion in meeting their requirements at the service station level have led seven manufacturers representing about 70 per cent of the industry production to ask that "no change" be made from the passenger car factory fill. These recommendations have been reinforced by the removal of the drain plug.

From your point of view this type of recommendation means a loss of sales of from 10-20 million pounds a year per model year at the service station level. From the point of view of the customer and the manufacturer, it means gears which are lubricated by oils contaminated with breakdown products, moisture, road abrasives, and metal particles as well as with somewhat depleted additives. What is the answer? How do we return to normal drain period recommendations? How can we avoid confusion at the service station level? Obviously the answer is a universal gear lubricant that is truly universal.

There has been rather good agreement on the philosophy that such oils are needed but the guesses on oils that would meet all requirements ranged from present multi-purpose oils to those which would meet the most difficult operating conditions which could be devised. After considerable debate, some of it rather acrimonious, progress began to be made.

First of all it was agreed that the logical place to start was to use the military specification oils as a base line. This specification incorporated the tests with which both the automotive industry and the petroleum industry are most well acquainted since they had been developed jointly by the two industries and the military. In addition to this, the Ordnance group had indicated a strong desire to have the military specification conform to what was available in the civilian market so as to simplify the supply problem in the case of an emergency.

Figure 9



With this general approach in mind, the problem fell logically into three phases:

1. To determine what level of performance the present tests were defining, in some absolute terms.
2. To establish several ranges of test activity which would describe all existing oils and allow a range for oil improvements.
3. To find out where in the range of test severity an oil must fall in order to meet present requirements.

Before going very far, it became apparent that both the L-19 and L-20 tests left something to be desired in the way of reproducibility. The L-20 condition was improved by closer control of the test gears. This was found impractical with the L-19 third members, but considerable improvement was brought about by closer attention to a variety of causes which tended to reduce accuracy. These were pointed out by Mr. H. R. Barton of Armour Institute and his associates, after studying a large number of L-19 tests. Mr. Barton also initiated a series of stress measurements under various conditions of L-19 operation. When these are completed the first quantitative information on the test severity of the L-19 will be available. Similar measurements are being made on the L-20, but here the problem is not so difficult since no change in loading is called for during the test.

At the same time it was felt that a series of test oils should be set up which could be used as standards of severity. This became a chicken and egg proposition, because in order to set up the oils, some quantitative idea of test severity had to be obtained, and the only available mechanism for doing this was to have a series of test oils. The idea was, therefore, temporarily abandoned.

Meanwhile the Ordnance department was conducting tests under what they considered to be their most severe conditions. The test schedule involving heavily loaded vehicles included operation simulating rugged cross-country travel followed by a trip through hot desert sands, which would emphasize high torque and low speed.

All vehicles operated with rated highway carried and towed loads. After break-in operation of 500 miles at speeds not in excess of 35 m.p.h., vehicles operated 4,500 miles on a relatively level section of Highway 95 at a maximum speed of 50 m.p.h. This constituted the first high speed phase of the test. All differentials were then inspected prior to departing for Death Valley, California for the high torque phase of the test. In the high ambient temperature of Death Valley and on the mountainous grades bordering the Valley, vehicles accumulated 2,000 miles each. Vehicles returned to Yuma Test Station at speeds not in excess of 35 m.p.h., and differentials were inspected to determine the effect of the high torque operations The second high speed phase of the test was accomplished on Highway 95. All vehicles accumulated a total of 10,000 miles each.

Taking the high speed and high torque phases and combining them into a single test, the CRC Group on New Test Techniques headed by Mr. T. P. Sands of Monsanto Chemical Company, found that they could reproduce the failures encountered in Ordnance tests, and have proposed the new test technique tentatively identified as L-37 to replace the L-20 high torque test. A point by point comparison of the two test procedures can be obtained from Figure X.

But what of the high speed tests? Since the first abortive attempt at establishing several oils which could be used as standards of oil and test performance, several materials had been developed which appeared to have the ability to react satisfactorily on the most severe high speed, high shock load test, yet which were also satisfactory for truck operation. Mr. T. P. Sands mixed one of these oils in various concentrations with base oil and set up a series with fifteen levels. The vehicle manufacturers then were invited to state their present and future requirements for high speed shock load resistance. Many tests are going on at present in order to set this severity level. Where it will fall is anybody's guess. Originally, it was felt that it would be around 12, the level of active sulfur. Since that time several manufacturers have found that by phosphate treating their gears as did my company, they can get by with levels of about 8, the level which appears to be required by the military. My own guess is that it will be somewhat under 10. Since the present level is about 5-6, this means that future universal or multi-purpose gear oils will have to have a higher

About the Author

Charles M. Heinen is assistant chief engineer of the materials laboratories, Chrysler corporation. He joined Chrysler in 1934 and through a Walter P. Chrysler scholarship, received his master's degree in chemical engineering from Michigan University in 1942. During World War II Heinen worked on the atomic bomb project at Oak Ridge. Prior to taking his present position, he was head of the fuels and lubricants laboratories and then assistant managing engineer of the chemical engineering laboratories. Heinen is chairman of the SAE fuels and lubricants technical committee.

FIGURE X

Comparison of Proposed L-37 and L-20 Tests

Requirement	*L-20*	*Proposed L-37*
Axle	5.83:1 Army ¾ Ton	5.83:1 Army ¾ Ton
Number of Runs/Test	1	2
Gear Coating	None	1 Phosphated 1 Uncoated
Break In Conditions		
1. Ring Gear Speed	62 r.p.m.	None
2. Ring Gear Load	6000 inch/lbs.	
3. Temperature	140° F.	
4. Time	20 minutes	
High Speed Conditions		
1. Ring Gear Speed		440 ± 5 r.p.m.
2. Ring Gear Load		9460 ± 150 inch/lbs.
3. Temperature		300° F. Max.
4. Time		100 minutes
High Torque Conditions		
1. Ring Gear Speed	62 r.p.m.	80 ± 1 r.p.m.
2. Ring Gear Load	32,311 inch/lbs.	41,800 ± 150 inch/lbs.
3. Temperature	200 - 250° F.	275° ± 3° F.
4. Time	30 hours	24 hours

level of high speed activity. Since gear oils are not a fast moving item, all factors affecting reformulation of your oils is a subject which should demand your immediate attention so that you will have as much "lead time" as possible.

You may have noticed that I spoke of a new high torque test to replace the L-20 but that I have not discussed a similar replacement for the L-19. Actually, there are a wide number of individual company tests which have been developed. Unfortunately until the reference oils have been tested and a general agreement reached as to the level required, I do not believe real progress on selection of a substitute L-19 will be reached. It is my own personal hope that eventual agreement is reached on a test that produces high speed scoring without the necessity for a bump test, since this type of operation is difficult to reproduce. Whatever the final choice, I do not feel that it will be difficult to arrive at, once the severity level is established.

Summarizing the situation we find:

1. That there is evidence to indicate that some gear oils which are generally designated as universal by the military and multi-purpose at the service station level are not adequate as far as:
   a. High speed activity
   b. Heat stability.
   c. Corrosive protection
2. That field tests are being conducted which will define the minimum level of both high speed and high torque performance required of gear oils by today's vehicles.
3. That good progress is being made in modifying procedures in order to define the necessary quality level of gear oils.

If this work is carried to a successful conclusion, it will be possible to formulate one oil for use at the service station level and for the military, for use in truck or passenger car service. This should result in the following benefits to the petroleum companies, the military, the manufacturers, and the general public:

1. Reduced costs of handling.
2. No chance of errors in lubrication of cars or trucks due to incorrect choice of products.
3. Greater availability of oils to the military in times of emergency.
4. Better lubrication of gears as a result of a return to the practice of field refill.

These then are some of the technical considerations which will affect what and how you market gear oils in the future. Working together I am certain the difficulties will be overcome and that the problems of gear lubrication will fade as have other problems which have faced our two industries. ■

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Iodine Value	6 maximum
Saponification Value	198 to 203
Average Molecular Weight	279 to 283
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Color 5 ¼" Lovibond	Max. 25Y/2.5R

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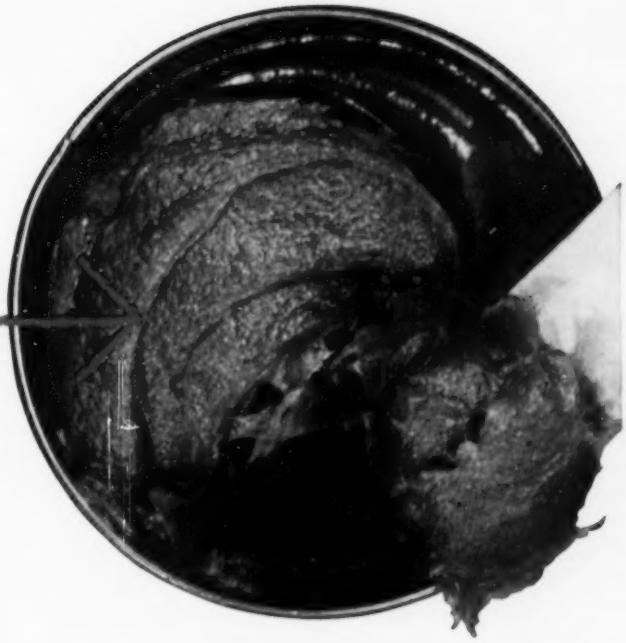

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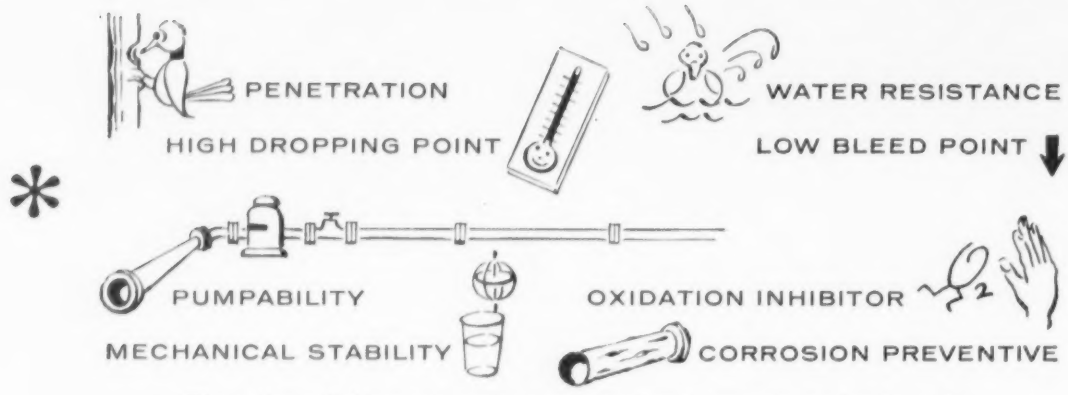

* The action of lithium as a gelling agent dominantly controls the first four of these functional properties. Fatty acids, oil and auxiliary additives influence the last four. Lithium does not adversely affect any of these critical properties.

There are definite manufacturing and marketing advantages in producing lithium-based grease. Inventory control is but one of them. Being a true multipurpose product, only one lithium soap grease is needed to lubricate your car: chassis, water pump, wheel bearings and universal joints. Often, several single-purpose lubricants are replaced in special industrial applications. Of equal significance is Lithium Corporation's acceptance in the field: over 10 years' production experience supplying lithium hydroxide to the grease industry. Add to this our unsurpassed production facilities and you are assured of a reliable source of consistently uniform quality LiOH, available from inventory for immediate shipment. Since we are equally interested in developing better lithium products, why not get in touch with us? A card or letter will bring immediate response.

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By Ray Ashworth

Northwestern University

THE SITUATION I wish to discuss has economic, social and political implications that make it one of the most serious problems facing our country today. The unplanned, uncontrolled, inadequately supervised growth of traffic is a disease that is already costing the nation thousands of lives and billions of dollars a year, bringing unspeakable human waste, turning city neighborhoods into slums, causing disillusionment in our judical system, and bringing about a steady leakage from our national resources at the rate of billions a year—money which could be going into things like health and education and housing and defense.

Our purpose is not so much to remind ourselves of the gravity of the problem as to seek courses of action which can be taken on various levels by members of a great industry, an industry which has a real stake in highway efficiency and traffic accident prevention. I propose to consider the problem from the enforcement side in particular.

I will start with what has become almost a cliche' in traffic safety, but it expresses a truth so simple it is

often overlooked. It is this: a traffic accident is never a random event but a caused occurrence. Therefore it can best be understood—and prevented—in terms of its causes. This is why, in order to have a really effective highway safety program in your community, you must first be able to pin down and analyze the cause-effect relationship that exists. You must be able to isolate some of the chain reactions that spell out death, injury, congestion, delay, and property damage. It is not enough to blame it on too much traffic, or the wrong kind of laws, or a particular group of drivers. Facts are your only hope. Real, solid, accurate facts, that dig behind the surface phenomena of accidents and congestion.

To begin with, you need important data such as where your accidents tend most to occur, and when. Then you must start answering the big question, why? What are the factors, for example, that can explain an excessive number of collisions at 21st and Main? Why does a new highway have a high accident rate?

Unfortunately, many communities are unable to answer these questions. They simply do not have the facts they need.

That brings us to one of the most important activities in traffic law enforcement that must be done by the police: the investigation of traffic accidents. There are right and wrong ways of going about this activity, and in too many cities, what is called investigation is really only a system of accident reporting, with little attempt to determine the real causes. The right kind of accident investigation calls for specialized techniques, modern equipment, adequate manpower, as well as effective police management. The result should be a steady stream of reliable data concerning accidents as they are happening—data that can answer the questions of when, where, and why.

Of course, the data will itself be meaingless until it is subject to classification and analysis: this calls for a police traffic records system that can sift out useful information, so that a police executive can say, for example, "Accidents are being caused by illegal left turns on Central avenue, particularly between the hours of five and seven p.m." or "We need more enforcement of the speed laws south of the curve on Highway 86."

This kind of information is needed not only for the police to carry out effective enforcement, but for the other agencies working for highway safety: the traffic engineers, the courts, the driver license authority, the safety council, the schools, the news media, and so on. All of these organizations should be working on the many-sided problem of accidents and congestion, yet their effort must be coordinated, and based on accurate information, the kind that is supplied by well-conducted accident investigation and field studies.

When you have discovered some of the things that are causing the trouble in your community, when you have revealed the most frequent and basic causes for the accidents and congestion you are experiencing, you will find one factor looming on the horizon; the human factor. Bad roads, inadequate signalling, mechanical failures—these things can play their part, but to control dangerous human behavior would be to eradicate more than nine-tenths of the problem. This is because, whether through lack of skill, knowledge, or motivation, people operate their motor vehicles on the road net in such a way as to cause death and injury to themselves and others. So while the accident problem is partly physical, it is mostly psychological and sociological.

This means that whoever can most directly and effectively bring about a change in the behavior of drivers can accomplish the most toward making your community a safer place to live in.

In the long run we need a reconditioning of community driving habits—a whole generation of drivers with safer patterns of skill, knowledge, and attitude. What are some of the ways we can bring this about?

Education is one answer. Education for safety, beginning in kindergarten and including a good high school driver training program. At the same time, the driver license authority has a responsibility, not only to withhold the driving privilege from those who are unfit for it but to check up on repeater violators, to see if their trouble stems from some correctable condition. This adds up to the driver improvement. Meanwhile, there is much that the public information media can do to maintain a favorable climate to safety.

These are ways of influencing driver behavior over the long haul. They are very important. But they are not enough. Unfortunately, there will always be a residue of irresponsible motorist behavior—enough to keep the ambulances and the wreckers busy. People are going to drive cars when they are drunk. They are going to drive recklessly. They are going to commit a number of unsafe acts, except as they recognize they can't get away with it. That is why we have enforcement, and why we cannot get along without it. No community has ever been known to bring about safety on the streets and highways without an adequate traffic law enforcement program.

In the city of Chicago, enforcement has gone up about 400% in the last five years, and the death rate has dropped sharply. In a recent month there were fewer traffic deaths in Chicago than ever before, in

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spite of the greater number of cars on the streets. I could quote statistics from cities throughout the country definitely showing the direct relation between accidents and law enforcement. In a relatively short time, enforcement can reduce the number of unsafe and disorderly acts involving motor vehicles in a given area. But traffic laws don't become an effective deterrent until they are actually enforced.

Let me make it clear that when I talk about enforcement I do not mean the "nail him and jail him" type of enforcement. True, there are some kinds of violators, such as drunken drivers—and particularly the repeaters—who will only respond to this kind of penalty. But a police enforcement program worthy of the name includes a whole variety of techniques, each to be applied accordingly to the situation. Enforcement is going on when a police officer says, "Sorry, sir, no left turn here. Try the next corner." Enforcement comes from the written warning, useful for minor or borderline violations. Here the citizen is informed, for instance, that he has been driving too fast, that there is insufficient evidence to make an arrest, but that he is being warned. He is given a copy of it and told it will be filed at police headquarters. Enforcement like this has been found to have a good effect on driver behavior over a period of time.

Then of course, there is the actual citation and arrest, with subsequent court action. Here again, there are a number of courses of action the court can follow. It can send violators to school, or put them on probation, or send them back to the Motor Vehical Department for re-examination to determine their fitness to drive. It can fine them or it can jail them. Traffic law enforcement can be a number of different things. The right course of action depends upon the circumstance.

But now we run into another problem. Violations are being committed at all hours of the day or night, in all parts of the street and highway system. You have only so much police manpower and equipment. You face the very important tactical question of how best to deploy this limited force in order to have the greatest effect. Obviously the answer is to apply enforcement pressure exactly where and when it is most needed. Without such a technique, enforcement would be so diffused as to lose much of its value.

That is why it is so important to have an accurate picture of your current accident situation. If you can step to a spot map that shows instantly which intersections have the highest accident rates, if you can open a file drawer and pick out immediately the eight collisions that took place at 21st and Main, last year, and see what kind of accidents they were, what times of day they occurred, and what violations were involved, then you are well on your way to using your resources most efficiently. You can draw a careful bead on your target and see a definite effect, rather than relying on the scattered effect of a shotgun blast. This is the kind of traffic law enforcement we need in a day when the nation's total of registered vehicles has gone above sixty million and when close to 40,000 Americans lose their lives in traffic every year. This is the kind of traffic law enforcement that will pay off.

If I have been talking about traffic in terms of urban areas, it is only by way of example. For while cities have shown some improvement during the last 20 years, our accident problem has now shifted drastically to small towns and rural areas. The national mileage death rate—a figure that has been dropping steadily for years—went up in 1956. This can be attributed directly to an increase in accidents on the open road. Three-fourths of all traffic deaths last year occurred in places classified as rural. This may give us our biggest problem in the years ahead.

Meanwhile, vehicle registration figures, which have about doubled in the last ten years, are still going up. This won't make the problem any easier. Given the present trends in population growth, and assuming we are not in for a severe depression, we can expect 80 million motor vehicles competing for space on the nation's streets and highways by 1975. You can see what this will mean simply in terms of the police problem, for instance, since most departments can-

Ashworth

About the Author

Ray Ashworth is acting director of the Traffic Institute and the IACP traffic division. He began his professional career with the Wichita police department in 1928, and was acting chief when he took leave to become a field representative and one of the original staff members of the Traffic Institute and IACP division. He has served as chief of police of San Antonio and Houston, Texas.

He served throughout World War II in Europe and the Pacific. Since that time he has been chief of the inspection and investigation division of the Veterans Administration regional office in Dallas, and chief of the public safety branch of the U.S. office of military government in Berlin.

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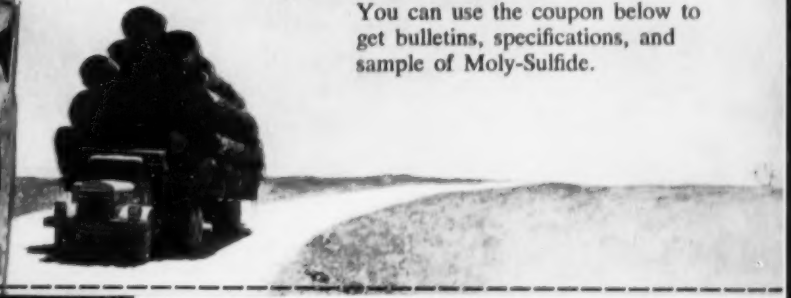

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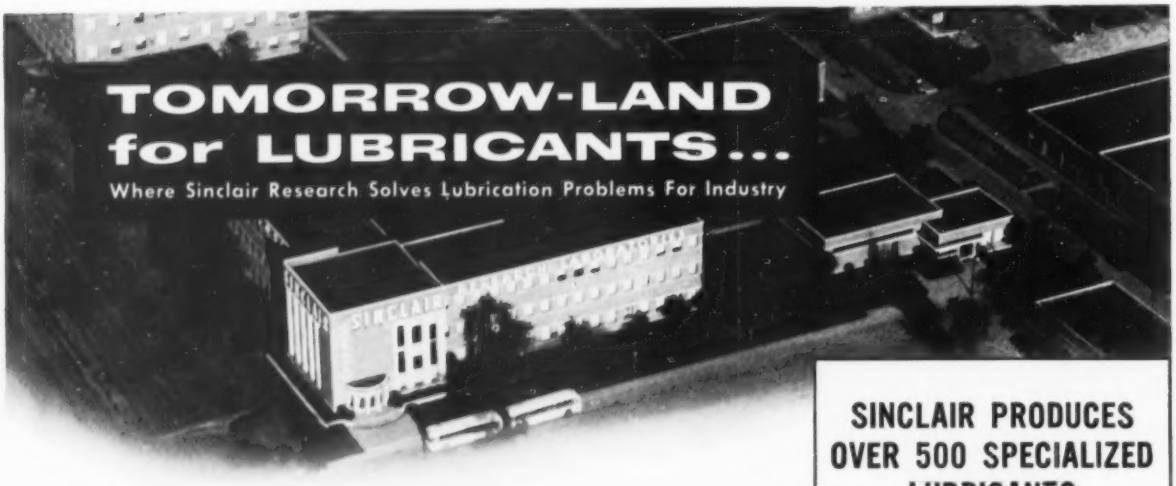
not hope to increase their budgets and manpower accordingly. It means that the administration of traffic police work, already a specialized activity, must be carried on with the most effective management techniques available. Methods of supervision and training must be as efficient as they need to be in any business. Only here the profit is counted in lives.

Clearly the job ahead can't be done unless officials who are responsible for traffic law enforcement receive enough modern, professional training—training in traffic police administration such as it offered by the Traffic Institute; training for judges and prosecutors and other traffic court officials, through active organizations like the American Bar Association. Specialized training is as necessary here as in operating a service station or running the Standard Oil Company. In traffic, however, there is an added sense of urgency, since the problem is growing as we watch it.

Two things become clear as we face the necessity of bringing about a decent and reasonably efficient system of highway transportation. First, it won't come about through some sudden discovery, some new law, some one organization stepping into the picture. There is no such simple answer to a problem that is at once physical, social, economic, and political. On the other hand, the solutions are not really mysterious or beyond our imagination, they are not as complex as in the field of crime, for instance. The solutions are in our hands today. They are evident in communities all over the country who have demonstrated their ability to find them. The steps which must be taken, the things we must do, have already been discovered, through experience and research. We now know that a coordinated safety program, working through such channels as education, enforcement, engineering, and driver license administration, and given the right kind of public support, can bring about really remarkable results in any community.

These things we know. Further, we believe that the problem of highway efficiency and traffic accident prevention is one with which a healthy democracy can successfully cope. This we must set about to prove. ■



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Patents and Developments

Carbon Black Concentrates

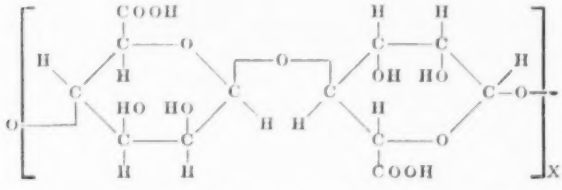
A method for producing pumpable carbon black concentrates (deliberately avoiding grease characteristics), mainly for production of highly radiant flames, is described in U.S. Patent 2,754,267 issued to Shell Development Company.

Lubricating Grease Containing a Metal Salt of Algin

Greases having desirably high dropping points and high mechanical stability to working have been prepared by using the alkali or alkaline earth metal soaps of combinations of high and low molecular weight carboxylic acids. It is also known that these greases require soap contents of at least 10% to exhibit the desired stability and freedom from oil separation. As the soap thickeners are diluted with lubricating oil, i.e. as the soap concentration of the grease decreases, the dropping point is materially lowered and semi-fluid products of poor structural stability result.

In U.S. Patent 2,754,268, Esso Research and Engineering Company reports that greases having excellent high dropping points and highly desirable structure and stability characteristics at much reduced soap requirements may be obtained by incorporating into the grease thickener at least a substantial proportion of a metal soap, particularly an alkali metal soap, of alginic acid. Preferably, the alginic acid soap forms all or part of the high molecular weight fatty acid soap constituent of a thickener of the high molecular weight-low molecular weight complex soap type discussed above.

Alginic acid, more commonly called algin, is a seaweed colloid usually extracted by alkais from brown algae (Phaeophyta) especially from members of the kelp order (Laminariales). The alginic acids are generally regarded as being hydrophilic colloidal polymers of anhydro-B-D-mannuronic acid units having the structural formula which is generally accepted to be:



It is not known whether the units have the pyranose or furanose structure, but the former is indicated in view of the excellent stability of alginic acid with respect to hydrolysis and the large negative rotation of the acid.

Natural algin in kelp is probably a combination of calcium alginate and aliphatic acid; it is insoluble in water and easily converted to a water solution of the sodium salt. In commercial preparation procedures, the kelp is easily digested with sodium carbonate or hydroxide, the resulting viscous liquor is separated from the residue, and alginic acid is precipitated by the addition of strong mineral acids. Since alginic acid is rather heat-unstable, whereas its salts or soaps are quite stable, it is generally marketed as sodium alginate. Various methods of producing algins are known to the art. A preferred procedure is disclosed in U.S. Patent 2,036,922. In general, polymers of anhydro-mannuronic acid and the stereo isomers thereof are suitable in the practice of the patent.

Some of the advantages of the patent may be realized by using metal soaps, particularly alkali metal soaps of an alginic acid as the sole or essentially sole thickener. Soap concentrations of about 2-15% should be used in this case. However, the alginic acid soap is present in the grease in the form of complex with a salt, preferably an alkali metal salt of a low molecular weight carboxylic acid. In many cases, it may be desirable to incorporate into the complex a soap of a high molecular weight fatty acid other than alginic acid. Thickeners of this type may contain about 10-75 wt. per cent, preferably about 25-50 wt. per cent, of algin soap. The low molecular weight acid salts and total high molecular weight acid soaps may be present in equi-molecular proportions. However, the low molecular weight acid may be present in smaller or larger proportions, mol ratios of 1:1 to 3:1 of low to total high molecular weight acids being preferred.

The total complex thickener content may vary within the wide range of about 2-40 wt. per cent of the finished grease. It is, however, a particular advantage of the patent that excellent greases having high dropping points and high structural stability to mechanical working may be obtained by using substantially less than 10 wt. per cent and even as little as 2.5 wt. per cent of an alginic acid soap containing-thickener. Complex soap concentrations of about 5-10 wt. per cent are, therefore, preferred for most purposes.

The low molecular weight acids to be combined with the alginic acid to form the complex soap thickener may be any aliphatic carboxylic acid having 1-5 carbon atoms per molecule, such as formic, acetic, propionic, butyric, valeric, furoic, acrylic acids, etc. The preferred low molecular weight acids are formic and acetic acids. High molecular weight fatty acids which may be used together with alginic acid and the low molecular weight acids here specified include saturated or unsaturated fatty acids having 10-30 carbon atoms per molecule, such as hydrogenated fish oil acids, stearic acid, oleic acid, behenic acids, etc.,

saturated acids having 18-22 carbon atoms per molecule being preferred.

Branched chain "Oxo" acids, prepared by the "Oxo" synthesis, are also useful as a component of the high molecular weight acids. In general, these acids may be prepared by any one of several well-known processes, such as by the catalytic reaction of an olefin with carbon monoxide and hydrogen at temperatures in the order of 300° to 400° F., and at pressures above about 1000 p.s.i., using cobalt carbonyl or other suitable catalyst. The resulting branched chain aldehydes or alcohols may then be oxidized or otherwise converted to the "Oxo" acids. In general, "Oxo" acids should be employed with other high molecular weight acids in concentrations below about 50% by weight based on the total non-alginic high molecular weight acid constituent.

Metal bases suitable for the formation of complex soaps include the hydroxides and carbonates of alkali, alkaline earth and certain other metals, for example, lithium, sodium, potassium, calcium, barium, and aluminum. However, alkali metal bases, particularly the hydroxides of sodium and lithium, which form complex soaps of satisfactory water insolubility and temperature characteristics, are preferred.

Greases in accordance with the patent may be prepared by preforming the alginic soap or soap complex with other acids and incorporating the same in the lubricating oil by mechanical mixing. The performed soap or soap complex may be prepared by neutralizing the alginic acid or mixtures thereof with the other acids to be used by means of substantially stoichiometric amounts of the desired metal base preferably in aqueous solution, recovering the complex soap formed and compounding the same with the lubricating oil at grease-making conditions.

However, the preferred method of the patent involves formation of the soap or soap complex in situ in the lubricating oil in the course of the grease-making process. For this purpose the alginic acid and other high molecular weight fatty acids, if any, may be charged to a reaction zone and heated together with a portion of the lubricating oil to about 130°-170°F. Thereafter the low molecular weight acid may be added and the mixture neutralized, preferably with a slight stoichiometric excess of an aqueous solution of the metal base of about 30-50% concentration. Sufficient metal base should be used to give the grease a free alkalinity of about 0.1-1% as NaOH.

The mixture may then be dehydrated at temperatures of about 250°-350°F. When dehydration is substantially complete, further lubricating oil may be added and heating continued to about 450-550°F. Thereafter, the grease may be cooled. When temperatures below 300°F. are reached, other conventional additives, such as anti-oxidants, particularly amino compounds, extreme pressure agents containing sulfur, halogen and/or phosphorus, etc., may be added in any conventional manner.

Example

Ingredients	*Weight Per Cent*
Alginic acid[1]	10.00
Hydrofol Acids 54[2]	10.00
Acetic acid (glacial)	4.00
Sodium hydroxide	6.50
Phenyl alpa-naphthylamine	1.00
Blend of naphthenic-type mineral oil distillates having a viscosity of 50 S.S.U. at 210°F.	68.50

[1]Commercial alginic acid which was a free-flowing fibrous powder.

[2]Hydrogenated fish oil acids corresponding in degree of saturation to commercial stearic acid.

Preparation

The alginic acid, Hydrofol acid and about ½ of the mineral oil were charged to a grease kettle and warmed to 150°-160°F. At this temperature the acetic acid was charged followed immediately by a 40% aqueous solution of sodium hydroxide. Heating was continued and at 300°F., when the soap mass in the kettle was fairly dry, the balance of the mineral oil was added. The mixture was further heated to about 500°F. At this temperature heating was discontinued and the mass cooled to 250°F., at which temperature the phenyl alphanaphthylamine was added. The grease was further cooled to 200°F., and homogenized by passage through a Gaulin homogenizer. Gaulin homogenization of the grease at higher rates of shear result in an excellent, smooth, uniform product.

Properties

Per cent free alkalinity as NaOH	0.72
Penetrations, 77°F. mm./10:	
Unworked	102
Worked, 60 strokes	114
Worked, 100,000 strokes (270 1/16" hole worker plate)	Too hard–did not run
Dropping point, °F.	500
Water washing test, per cent loss	None
Norman Hoffmann oxidation test, hours to 5 p.s.i. drop in O^2 pressure	210

Wide Temperature Range Alkali Metal Grease Containing Excess Alkali Metal Hydroxides

U. S. Patent 2,755,247, issued to The Texas Company, discloses ball and roller bearing greases which are characterized by high dropping points and outstanding performance over a wide temperature range. The novel alkali metal greases are suitable for operation at temperatures as low as −20°F. and as high as 500°F., and higher.

They result from elevated temperature digestion of a mixture comprising mineral oil, alkali metal soaps of a fatty material of prescribed composition, and a critical amount of excess alkali metal hydroxide. The critical factors in the production of the wide temperature

range greases of this patent are a critical quantity of excell alkali metal hydroxide present during the high temperature digestion, the composition of the fatty material from which the alkali metal soaps are formed, the temperature and duration of the high temperature digestion, and rapid chilling plus milling of the drawn grease.

The greases are prepared by digesting at a temperature of 480 to 550°F. for a period of 2 to 5 hours a dehydrated reaction mixture comprising 15 to 25 per cent alkali metal soaps of a fatty material having an iodine number of at least 90, 60 to 80 per cent mineral oil and 1.7 to 2.3 per cent alkali metal hydroxide. After digestion at the prescribed temperature, additives, such as aluminum stearate, which imparts water resistance, are incorporated in the grease mixture which is then stirred for an additional period of 20 minutes to 60 minutes prior to drawing at a temperature of about 500°F. The drawn grease is rapidly cooled in pans or in a rotary chiller whereby the grease is converted to a hard grease. Milling of the rapidly cooled hard grease in a roller mill or in a colloid mill produced smooth, glossy, hard greases which are characterized by dropping points over 500°F., good oxidation resistance and excellent low temperature torque properties.

The alkali metal greases of this patent are said to be important additions to the field of commercial greases. The high dropping points and good low temperature torque properties of the alkali metal greases adapt them for use over a wide temperature range. The sodium base greases are particularly useful commercial greases. Sodium base greases of the composition prescribed are smooth, glossy greases with dropping points over 500°F. The preparation of alkali metal greases characterized by the aforesaid properties represents a significant advance in the art of grease making.

Naphthene base oils or paraffin base oils can be employed to prepare the novel alkali metal greases. With the paraffin base oils it is necessary to incorporate petroleum mahogany sulfonate salts as stabilizing agents in order to obtain suitable greases. Sodium petroleum mahogany sulfonate is ordinarily employed as the stabilizing agent. With paraffiin base oils the added petroleum mahogany sulfonate salt amounts to about 0.25 to 4 per cent of the final calculated grease composition. The sodium petroleum mahogany sulfonate is added to the reaction mixture together with the paraffin base oil and is present during saponification and dehydration of the reaction mixture. When naphthene base oils, commonly known as pale oils, are employed, it is not necessary to employ stabilizing agents such as petroleum mahogany sulfonate salts since greases of high stability are prepared without the use of a stabilizing agent.

The mineral oil content of the greases of this patent comprises 60 to 80 per cent of the total calculated grease composition. Advantageously, the mineral oil content comprises approximately 65 to 75 per cent of the total grease composition.

Soap-forming materials possessing a substantial olefin content are required for the formation of the alkali metal greases. The soap-forming stock, which can be soap-forming fatty acids, fatty esters or mixtures thereof, should possess an iodine number of at least 90 and preferably between 90 and 130.

A particularly preferred soap-forming material comprises a mixture of menhaden oil and hydrogenated fish oil fatty acids, which latter are commercially known as Snodotte acids. A 3 to 1 menhaden oil-Snodotte acid mixture having an iodine value of approximately 110 has proven particularly useful as a soap-forming stock.

Commercially available Snodotte acids have approximately the following soap composition:

	Per Cent
Myristic acid	9
Palmitic	22
Stearic	27
Arachidic	22
Behenic	20

Typical tests on commercially available Snodotte acids are as follows:

Sap. No	197
Neut. No	193
Iodine No	1
Titer, °C	54.6

Estersil-Thickened Greases

According to the teaching of U. S. Patents 2,657,149 and 2,676,149, an estersil is an organophilic solid in a supercolloidal state of subdivision, having an internal structure of inorganic siliceous material with a specific surface area of at least 1 m.²/g., having chemically bound to said internal structure–OR groups, wherein R is a hydrocarbon radical in which the carbon atom attached to the oxygen is also attached to at least one hydrogen atom, each –OR group having from 2 to 18 carbon atoms.

In U. S. Patent 2,746,922 issued to E. I. duPont de Nemours and Company, it is demonstrated that the rheological properties of lube oil-estersil compositions may be controlled by incorporation of a hydrogen-bonding electron donor compound in which no electron donor atom is attached to two different silicon atoms.

It has been found that by mixing polyfunctional hydrogen-bonding donor compound, such as water, ethylene glycol, and glycerine, with a water-in-soluble oil, such as a hydrocarbon oil, and an estersil, the thickening efficiency of the estersil can be improved to produce a grease which has a substantially more constant consistency upon storage under humid conditions. By adding a hydrogen-bonding donor compound such as tricresyl phosphate and di-n-butyl ether, in

Continued on page 36

1957 NLGI COMMITTEES

With the listing of the 1956-57 committees below it becomes apparent that not only has the number of committees grown (twelve this year as opposed to five in 1951), but that greater scope of services and more activities are in the offing for the National Lubricating Grease Institute. The twelve group headings shown here are part of the broadening of industry effort and include special internal projects designed to continue the pattern of improvement within the Institute. As NLGI approaches the quarter-century mark since its foundation, the organization, promises to continue in its tradition of service to the industry, thanks to the efforts of these men and those who have served before.

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Technical Committee

Chairman T. G. Roehner, Director of the Technical Service Department, Socony-Mobil Laboratories

Plans for the activities of the NLGI Technical Committee for the October 1957 meeting are already taking definite shape. It has been decided that the committee will again undertake organization of a panel which will be given a half day of the first two days of the main meeting. The 1957 subject will be "Lubricating Greases for Contractors Equipment." Discussions during the past meeting with a number of members left no doubt that there is widespread interest in this subject. Furthermore, numerous opinions were expressed to the effect that the subject is timely. As in preceding sessions of this type, the panel will be made up of representatives from manufacturers of contractors' equipment, grease dispensing manufacturers, lubricating grease manufacturers, and of the customer for all three industries, the contractor.

It was also agreed at the 1956 Annual Meeting that a symposium would be included on the agenda for the 1957 Technical Committee meeting. Strong support has been received for bleeding of lubricating greases as the subject. The objective will be to devote more time to the fundamental side of the subject rather than to the so-called practical aspects, although the latter will not be neglected.

Committees to organize these two sessions have not yet been finalized, and, therefore, any suggestions regarding membership would be appreciated. Recommendations regarding speakers would also be helpful and should be sent to Messrs. L. C. Brunstrum, E. W. Nelson or T. G. Roehner.

Hoyst Ledpantz

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which the donor atom has a relatively weak tendency to donate electrons, to lubricants comprising a hydrocarbon oil and an estersil, the tendency for such lubricants to form a gel-like mass upon storage at elevated temperatures also can be reduced. Further, by adding a monofunctional hydrogen-bonding donor compound, such as stearyl alcohol, stable dispersions of estersils in hydrocarbon oils can be prepared, such dispersions being particularly useful because of their improved viscosity index.

Certain estersil-thickened greases have a tendency to harden between intermittent periods of use and, when in this hardened condition, the film formed by such a grease is rather easily cracked and broken. Corrosive agents such as air and moisture are enabled to reach the metal surface through such cracking to thereby corrode it. Through proper rheological control effected by adding a hydrogen-bonding donor compound, especially the polyfunctional-type compounds, it is possible to reduce substantially such hardening and consequently minimize film failure and resultant corrosion.

Reduced gelation characteristics of estersil-thickened greases modified with relatively weak electron donor compounds according to the patent results in improved feeding characteristics when such greases are used for lubricating roller-type anti-friction bearings. This advantage is particularly noticeable when such bearings are used under high loads.

The advantage of using a monofunctional hydrogen-bonding donor compound for controlling rheological properties of a lubricating oil is especially pronounced in situations where a relatively minor proportion of an estersil is used to improve the viscosity index of a lubricating oil. For instance, by adding the monofunctional hydrogen-bonder, stearyl alcohol, to a dispersion of 2% of an estersil in a hydrocarbon oil, one can maintain a satisfactory suspension of the estersil in the oil over a considerably increased period of time.

Other important advantages of rheological control according to the patent are observed when estersil-thickened greases are used on combination with other agents commonly encountered under conditions of practical operation. Thus, when bearings may be lubricated alternately with conventional soap-thickened greases and with greases thickened with an estersil, consistency changes in the estersil-thickened grease may occur, and it is possible to control such changes by the addition of suitable hydrogen-bonding compounds. Similarly, various conventional additives are used to impart particular properties to lubricating oils and greases made from such lubricating oils, and by the use of suitable hydrogen-bonding donor compounds one can control the rheological properties of the resultant mixtures as desired for particular uses.

The Estersil

Estersils suitable for use in the compositions of the patent may be prepared by esterifying an inorganic siliceous material having a specific surface area of at least - $m^2./g.$ with a primary or secondary alcohol in which the hydrocarbon radicals have from 2 to 18 carbon atoms. While the siliceous material which acts as a substrate for the estersil may have a specific surface area of as little as 1 $m^2./g.$, it is preferred to use estersils in which the substrate has a specific surface area of at least 25 $m^2./g.$

To make an estersil, one takes a substrate of particles longer than colloidal size having a surface of silanol (SiOH) groups and esterifies it under substantially anhydrous conditions with a primary or secondary alcohol having from two to eighteen carbon atoms in its structure. The substrate thus acquires a coating of –OR groups bound to the silica surface, and, since the alcohol used was primary or secondary, the particular atom in the –R chain attached to the oxygen atom will also necessarily be attached to at least one hydrogen atom. The substrate particles are of such size and character that they have a specific surface area of at least one square meter per gram—that is, either the individual particles are solid, dense, siliceous material so finely divided that the surface area is in this range, or the particles are larger but so porous that the surface areas is thus extended.

Suitable substrates for making into estersils may be in any of a wide variety of forms. Although the surface must be silica, the substrate need not be silica throughout its structure, and materials, upon the surface of which silica has been deposited or otherwise effected to be present, are entirely suitable. Particles of clay, talc, asbestos, mica, and other silicate minerals, for instance, have some surface silanol groups and may be esterified to form estersils, or the proportion of surface silanol groups may be increased by depositing amorphous silica thereon or by treating the silicates with an acid, such as sulfuric, to dissolve our metal ions and leave more silanols.

Preferred as substrates for some uses, however, are amorphous silica products, especially porous, coherent aggregates of ultimate, amorphous silica particles. Silica gel has such a structure and may be used as a substrate. Open-pored substrates of this general type, wherein the pores have an average diameter of at least four millimicrons, can be esterified and then dried, preferably from an organic liquid, to give estersils which are very porous and easily crushed by mechanical action. The structure of the aggregates in the substrate may be reinforced, prior to esterification, by mixing them with an aqueous dispersion of active silica and heating above 60° C. at a pH of 8 to 11, whereby the active silica accretes to the aggregates.

The ultimate silica units in the aggregates of substrate need not be the 2 to 5 millimicron particles present in a conventional silica gel. Rather, they may be built-up particles, such as those of Patent 2,574,902. For instance, a sol prepared by ion exchange as described in Patent 2,244,325 may be heated above 60° C. and further quantities of the same type of sol

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may be added with continued heating until at least five times as much silica has been added as was originally present, whereby the ultimate silica particles are built-up into the size range of 15 to 130 millimicrons. These particles may be coalesced, as by adding a long-carbon-chain nitrogen compound as described in Patent No. 2,663,650. Alternatively, ultimate silica units may be up in the presence of salts by adding an acid, such as sulfuric, to a sodium silicate solution above 60°C. at a sodium ion normality not over one, over a period of time, or by adding to a silica sol above 60°C. a silicate solution and enough acid to maintain the pH at from 8 to 11, and continuing the additions until the ultimate units have reached the desired size, as described in Patent No. 2,601,235.

The alcohol used for esterifying the substrate may be any primary or secondary alcohol having at least two carbon atoms. It may be straight– or branched–chain, or alicyclic, saturated or unsaturated, alkyl or aromatic. Examples of alcohols which may be used are: ethyl, n-propyl, n-butyl, n-pentyl, n-hexyl, n-heptyl, n-octyl, n-nonyl, n-decyl, n-undecyl, n-dodecyl, n-tetra-decyl, n-hexadecyl, n-octadecyl, isobutyl, isoamyl, 2, 2, 4-trimethyl hexane-1-ol, 5, 7, 7,-trimethyl 2-octaine-1-ol, isopropyl, sec-butyl, sec-amyl, sec-n-octyl, methyl iso-butyl carbinol, di-iso-propyl carbinol, cyclopentanol, cyclohexanol, cycloheptanol, menthol, allyl, crotyl, oleyl, citronellol, geraniol, propargyl, benzyl, beta-phenyl-ethyl, hydrocinnamyl, alphamethylbenzyl, and cinnamyl.

To effect esterification, the alcohol is heated with the substrate under substantially anhydrous conditions. Water formed by the reaction is removed, as by azeotropic distillation. Usually, an excess of the alcohol over that theoretically required is employed, and the water content of the alcohol at the end of the reaction period should not exceed about 5% for maximum esterification. The rate of reaction increases as the temperature is raised, suitable temperatures to give the indicated type of product in a one-hour reaction time being as follows:

	Primary Alcohols, °C.	*Secondary Alcohols,* °C.
Organophilic	100	130
Hydrophobic	118	225
Maximum esterification	190	275

The esterfied products are ogranophilic in that in a two-phase n-butanol-water system, for instance, they prefer to be wet by the butanol. When sufficiently esterified, they are hydrophobic–that is, are not readily wetted by water. When esterified to the maximum degree, they are impervious to methyl red dye and do not adsorb it from anhydrous benzene solution. The –OR groups in the estersil are chemically reacted with the silica surface and cannot be removed by washing with hot methyl ethyl ketone or similar solvents, or by prolonged extraction in a Soxhlet extractor. No

alcohol is displaced from the silica by treatment with solvents.

The properties of estersils are related to the number of ester groups per unit surface area of the substrate. Although the surface-modifying characteristics of different –OR groups varies considerably, as estersil is generally organophilic when there are present on the surface more than about 100 ester groups per 100 square millimicrons of substrate surface area. When this proportion is 200 or more ester groups per 100 square millimicrons of substrate area the products are generally hydrophobic, and when it is 270 or more, the products do not adsorb methyl red dye from anhydrous benzene solution. On an unknown estersil, the –OR group can be identified by decomposing the estersil with acid to release the alcohol, and identifying the alcohol by usual analytical methods. The substrate surface area can be measured by burning off the ester groups in a stream of oxygen at 500°C.

The Lubricating Oil

The oils used in making the compositions of this patent are water-insoluble lubricating oils. Oils which contain, say, 5 to 10% of a water-soluble component, or which are themselves soluble to that extent in water, can be classed as essentially water-insoluble oils. However, to obtain maximum advantages of the water resistant properties of the estersils used, the oil should have as low a solubility in water as possible and preferably should not be soluble in water to the extent of more than about 1%.

A wide variety of oils can be used. In general, any water-insoluble animal, vegetable, or mineral oil or synthetic chemicals having typically oily characteristics and lubricating or friction decreasing properties can be used.

Illustrative of suitable water-insoluble lubricating oils are: hydrocarbon oils such as naphthene base oils, paraffin base oils, and petrolatum; silicone oils, such as the dimethyl siloxane linear polymers; florocarbon oils such as the perfluorinated petroleum oils; vegetable oils such as cotton seed oil and castor oil; animal oils such as sperm whale oil, lard oil, blow fish oil and degras; and water-insoluble synthetic chemicals having typical oily characteristics such as di (2-ethyl hexl) adipate, bis-nonyl glutarate, di (2-ethyl hexyl) thiopropionate, di (2-ethyl hexyl) oxydibutarate, propylene oxide-tetrahydrofuram copolymer, di (2-ethyl hexyl) sebacate; and dimethyl cyclohexyl phthalate.

The Hydrogen Bonder

According to the patent a hydrogen-bonding donor compound is added to an estersil-lubricating oil composition of the type just described to effect control of the rheological properties of the mixture.

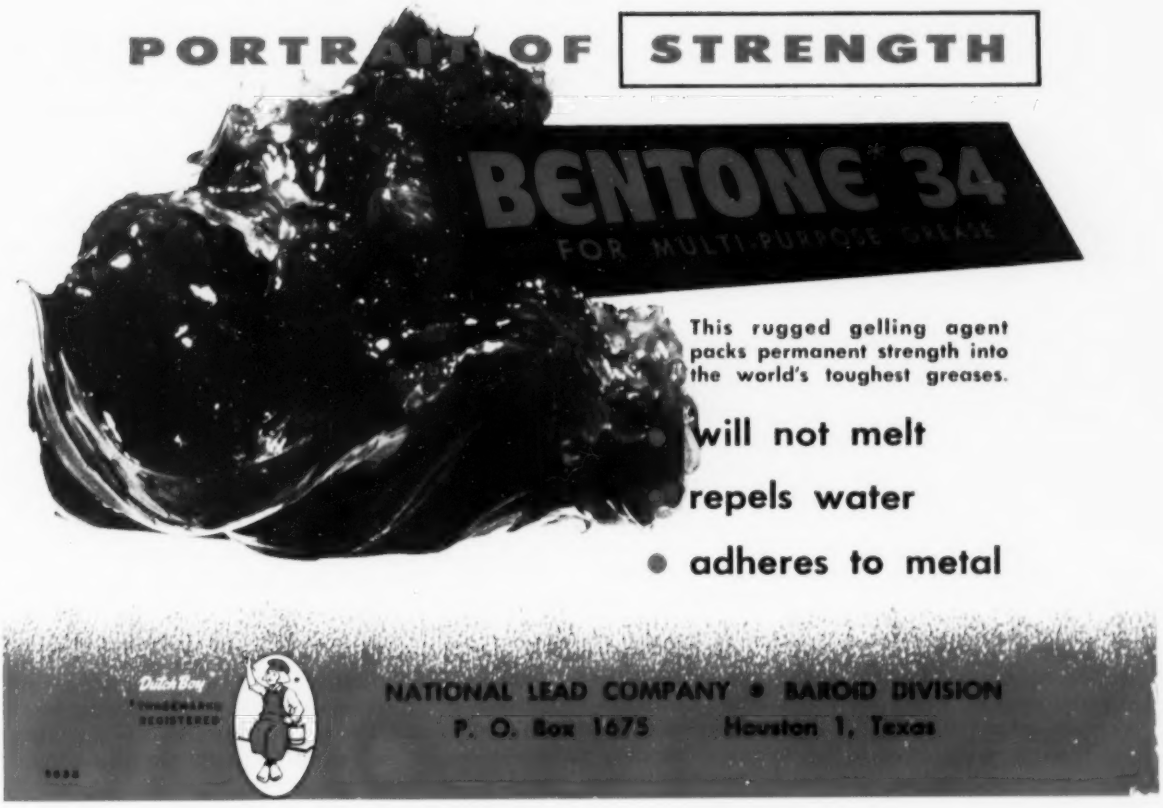
Hydrogen-bonding donor compounds are a well recognized group of chemical compounds having a certain electronic configuration. Hydrogen-bonding is a concept advanced in recent years to explain abnormalities in the chemical and physical behavior of mixtures of compounds one of which contain hydrogen attached to a strongly negative radical and the other an atom capable of donating a pair of electrons to form a directional or coordination bond. Since the bond is formed by the donation of an electron pair from one atom, the donor, to the other atom, the acceptor, the bond is not of the type conceived of as an ordinary valence bond but many of the properties of the mixture indicate that a real association between the donor and acceptor molecules has occurred. These mixtures, for instance, exhibit an abnormal vapor pressure lowering, that is, a deviation from Raoult's law. There are further observed abnormal heats of mixing and abnormal deviation in viscosity and freezing point lowering.

It will be observed that the hydrogen-bonding donor compounds which are effective in the compositions do not necessarily function exclusively by reason of their character as hydrogen-bonding donor compounds. Certain classes, such as amines, ethers, esters and amides, probably function as donors because they contain a strongly electronegative atom which is capable of donating an electron pair to the hydrogen of a silanol group which may be present on the surface of an estersil. On the other hand, some of the electron donor compounds, such as water, organic hydroxy compounds and carboxylic acids, are capable of acting also as electron acceptors in that they contain a hydrogen capable of accepting an electron pair and such an electron pair may be donated from the oxygen of a surface silanol or siloxane group on the estersil particle. Suffice it to say that the compounds which by conventional methods, can be shown to be electron donors in the usual sense of the word, are effective. It may be further observed that some of the most highly effective electron donor compounds in the compositions are those which are capable of also acting as electron acceptors.

It seems quite likely that such compounds as water, alcohols, and carboxylic acids may function both as donor and as acceptor compounds in view of their high degree of effectiveness. In any event, it is believed that these materials would not function as acceptors to the exclusion of their function as donors.

From the above description it will be apparent that hydrogen-bonding donor compounds as a class may be used in the estersil-lubricating oil compositions of the present invention. The donor compound may be organic and of this group the ethers, amides, ketones, esters of phosphorus oxy-acids, and alcohols, as disclosed in Patent 2,392,767 may be employed.

If, in a compound, an atom capable of acting as an electron donor is attached to two different silicon atoms, as for example the siloxane oxygens of the polysiloxane or silicone oils, the compound has effects other than control of rheological properties when added to an estersil-oil composition. Hence, if the siloxane oxy-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gens of the silicone oils are considered to make these oils hydrogen-bonding donor compounds, such compounds are beyond the scope of the present patent and not included.

More particularly, donor compounds which are especially effective for controlling rheological properties in a manner which will increase the thickening efficiency of the estersil and minimize hardening of the grease upon storage, are polyfunctional compounds. Such polyfunctional compounds may be selected from the group consisting of polyhydroxy compounds such as ethylene glycol, glycerine, pentaerythritol, sorbitol, mannitol, dextrose, resorcinol, 4,4'-dihydroxy diphenyl, ricinoleyl alcohol, decamethylene glycol, 1, 3-propanediol, and propylene glycol; polyfunctional amines such as hexamethylene diamine and decamethylene diamine; polybasic acids such as oxalic, succinic, and azelaic; amino alcohols such as triethanol amine, 2-amino-2-methyl-1, 3 propane diol, and ortho- and para-amino phenol; alpha-amino carboxylic acids such as aspartic acid; hydroxy-carboxylic acids such as 12-hydroxy stearic acid, 9- 10-, 12-hydroxy stearic acid, and lactic acid; and other polyfunctional hydrogen-bonding donor compounds.

Particularly desirable to control rheological properties so as to reduce the tendency for gelation to occur, are hydrogen-bonding donor compounds selected from the group consisting of phosphoric and phosphorous acid esters such as tricresyl phosphate, tributyl phosphate, and tributyl phosphite; oxy- and thio-ethers such as di-n-butyl ether, "Lorol" methyl sulfide, dodecyl di-phenyl ether, "Lorol" sulfide, di-phenyl ether, and ethyl propyl sulfide; fatty acids esters such as stearyl palmitate, and sorbitan monooleate; condensation products of aromatic aldehydes such as benzion; and other relatively weak hydrogen-bonding donor compounds.

Particularly desirable for controlling rheological properties so as to partially or completely peptize the thickening structure of the estersil to cause the lubricating compositions to lose consistency in a controlled manner or to prepare stable dispersions of estersils in lubricating oils or hydraulic fluids, are hydrogen-bonding donor compounds from the class consisting of monofunctional alcohols such as stearyl alcohol, cetyl alcohol, and hydro-abietyl alcohol; monofunctional amines such as dodecyl amine, octadecyl amine, and "Lorol" amine; monofunctional amides such as steramide; certain metal salts of carboxylic acids such as zinc laurate, aluminum stearate, lead oleate and lead naphthenate, monoglycerides such as monoglyceryl stearate.

Water is a specifically perferred hydrogen-bonding donor compound for use in the present patent. It acts in a manner similar to ethylene glycol, glycerine, and

other polyhydrated compounds described above. It is, of course, unique in its structure in that its atomic configuration permits it to act readily either for donating a pair of electrons from the oxygen of the water to the hydrogen of a surface silanol on the estersil, or for accepting a pair of electrons from the oxygen of a surface silanol or siloxane grop in the estersil through the hydrogen of the water. Furthermore, water can act as a "bridging" agent in that the behavior just described can take place within a group of water molecules. Thus, the water can act in the same manner as a difunctional alcohol and cause an increase in the thickening efficency of the estersil. The grease so produced also has substantially more constant consistency upon storage under humid conditions and, as a consequence, permits improved rust prevention.

It will be apparent that mixtures of different hydrogen bonders may be employed in a grease and that, depending upon the character of the individual bonders, a combination of results can be effected. Thus, a mixture of equivalent amounts of octadecyl amine and a dibasic acid are quite effective in increasing the rust prevention characteristics of the grease without an accompanying loss of water resistance.

Conversely, even when a single hydrogen bonder is employed, the bonder may have a combination of properties some of which are desirable and others of which are either only tolerable or definitely undesirable. Thus, it is preferred to use hydrogen-bonding donor compounds which are nearly neutral, since acids or bases tend to reduce the water resistance of estersil-lubricating oil compositions.

Similarly, it is preferred to use compounds which are not too volatile—that is, compounds boiling above about 60° C. at atmospheric pressure in order that the effectiveness of the bonder may be retained after prolonged storage of the lubricant. For instance, diethyl ether is a hydrogen-bonding donor compound, but it is so volatile that its use would present problems during storage.

Again, it is preferred, in general, to employ as hydrogen-bonding donors, compounds which have a relatively high proportion of functional groups in proportion to the total molecular weight. Thus, water having a molecular weight of 18 contains at least 1 and potentially 3 functional groups—that is, groups capable of donating a pair of electrons or accepting a pair of electrons and is hence highly effective in small proportions. On the other hand, decamethylene diol, for instance, contains 2 functional groups at opposite ends of a 10 carbon chain and hence a larger weight percentage of the latter compound would be required to produce the same effect as a given proportion of water.

The hydrogen-bonding donor selected should be one which in the proportions used forms a homogeneous mixture with the estersil-oil composition. The mixture should be free of undispersed particles of the hydrogen-bonding donor compound. Thus, preferably the bonder is a liquid or is soluble in the oil to give a liquid product. In some instances, homogeneity may be achieved by heating the mixture of oil and hydrogen-bonder to elevated temperatures. Accordingly, a prospective hydrogen-bonder is not ruled out for lack of homogeneity in the mixture until it has been tested in the mixture at elevated temperatures.

Lubricants of controlled rheological properties are made according to the present patent by mixing the estersil, the lubricating oil, and the hydrogen-bonding donor compound. Any means adapted to provide effective mixing under the conditions present in the three-component system may be employed.

The mixing can be carried out in milling and mixing devices of the kind used heretofore for introducing other non-soap thickeners into oil. Paint mills, ink mills, colloid mills, ball mills, homogenizers, mixers of the sigma-arm type, and similiar devices can be used to give the desired thorough dispersion of the estersils in the oil. Provision for heating during mixing may be made. The type of milling or mixing device used to carry out the dispersion depends upon the physical and chemical characteristics of the particular estersil employed. Thus, an ink mill gives very good results in most cases, whereas a homogenizer which produces high shearing forces gives maximum dispersion and thickening efficency particularly in the thinned greases. The power requirments of the mixing operation are low, especially with estersils having an amorphous silica substrate because of the organophilic character and porous, pulverulent structure of the estersils.

It should be noted that, in the case of greases, the milling will be so intensive as to cause some break-up of the structure of the estersil. If the estersil is composed of aggregates or agglomerates of dense, ultimate, particles such aggregates of agglomerates may be broken down into the ultimate units or groups of them such as chains, rods, or sponge-like networks. Such milled fragments are, of course, still estersils since they contain surface silanol groups esterfied with primary or secondary alcohols.

The proportions of estersil, lubricating oil and hydrogen-bonder in a composition of this invention may be widely varied depending upon the properties desired in the finished product. For lubricating compositions, the lubricating oil is the predominant component and the estersil and hydrogen-bonder are minor components as to proportions. The estersil is highly effective as a thickening agent for the lubricating oil to make greases, a surprisingly small proportion of estersil being sufficient for this purpose.

The proportion of estersil used as a thickening agent depends upon such factors as the character and viscosity of the oil, the nature of the thickened oil desired, and the nature of the estersil itself.

The thickening efficiency of an estersil is determined by a number of factors, such as particle shape, interfaction of the surface of the particles with the oil,

FUTURE MEETINGS of the Industry

JANUARY, 1957

8-10 Kentucky Petroleum Marketers Association (annual meeting), Brown Hotel, Louisville.

17-18 National Industrial Conference Board (general session), Hotel Commodore, New York City.

21-25 American Institute of Electrical Engineers (1957 Winter general meeting), Hotel Statler, New York City.

23-24 Northwest Petroleum Association, Nicollet Hotel, Minneapolis.

FEBRUARY, 1957

8-9 Symposium on Composition of Petroleum Oil ASTM (Committee D-2), Jung Hotel, New Orleans.

22 Natural Gasoline Association of America (Permian basin regional meeting), Scharbauer Hotel, Midland, Texas.

24-28 AIME Annual Meeting, Hotels Roosevelt and Jung, New Orleans.

26-27 API Division of Marketing (Lubrication Committee), Sheraton-Cadillac Hotel, Detroit.

MARCH, 1957

11-12 Illinois Petroleum Marketers Association (35th annual convention), Hotel Pere Marquette, Peoria, Ill.

14-16 Texas Oil Jobbers Association (annual convention), Rice Hotel, Houston.

19-21 Ohio Petroleum Marketers Association (Spring convention and trade exposition), Deshler-Hilton Hotel, Columbus.

20-22 API Division of Production (Southern District Meeting), Washington Youree & Captain Shreve Hotels, Shreveport.

25-27 Midwest Gas Association meeting, Hotel Nicollet, Minneapolis.

27-29 American Power Conference meeting, Sherman Hotel, Chicago.

28 National Industrial Conference Board (general session), Sheraton-Palace Hotel, San Francisco.

APRIL, 1957

7-12 American Chemical Society (131st national meeting), Miami, site unreported.

10-12 API Division of Production (Mid - Continent District meeting), Mayo Hotel, Tulsa.

16-18 National Petroleum Association, Cleveland, Ohio.

18 National Industrial Conference Board (meeting of board), Waldorf - Astoria Hotel, New York City.

24-26 National Gasoline Association of America (36th annual convention), Rice Hotel, Houston.

24-26 API Division of Production (Rocky Mountain District meeting), Gladstone, Townsend & Henning Hotels, Casper, Wyo.

28-30 Independent Petroleum Association of America (midyear meeting), Buena Vista Hotel, Biloxi.

29-May 1 American Oil Chemists' Society (annual meeting), Roosevelt Hotel, New Orleans.

MAY, 1957

1-3 API Division of Production (Eastern District meeting), William Penn Hotel, Pittsburgh.

6-8 API Annual Pipe Line Conference, Cleveland Hotel, Cleveland.

13-16 API Division of Refining (midyear meeting), Sheraton, Hotel, Philadelphia.

15-17 Fuel Oil Distributors Association of New Jersey (annual convention), Hotel Berkeley, Carteret, Asbury Park, N. J.

16-17 National Industrial Conference Board (general session), Waldorf - Astoria Hotel, New York City.

19-22 Texas Independent Producers & Royalty Owners Association (annual meeting), Galvez and Buccaneer Hotels, Galveston.

22-24 API Division of Production (Eastern District meeting), William Penn Hotel, Pittsburgh.

JUNE, 1957

9-14 API Division of Production (midyear committee conference), Muehlebach Hotel, Kansas City.

10-12 Interstate Oil Compact Commission (midyear meeting), Canyon Hotel, Yellowstone National Park.

16-21 ASTM, Chalfonte-Haddon Hall, Atlantic City, N. J.

24-25 Michigan Gas Assn. meeting, Grand Hotel, Mackinac Island, Mich.

24-28 American Institute of Electrical Engineers (1957 Summer general meeting), Sharaton-Mt. Royal Hotel, Montreal.

SEPTEMBER, 1957

11-13 National Petroleum Association (55th annual meeting), Traymore Hotel, Atlantic City, N. Y.

30-Oct. 2 American Oil Chemists' Society (1957 Fall meeting), Netherland Plaza Hotel, Cincinnati.

OCTOBER, 1957

1-2 Texas Mid-Continent Oil & Gas Assn. (38th annual meeting), Texas Hotel, Fort Worth.

6-9 AIME (Petroleum Branch meeting), Dallas. Site unreported.

7-9 American Gas Assn. (annual convention), Kiel Auditorium, St. Louis.

7-11 American Institute of Electrical Engineers (1957 Fall general meeting), Morrison Hotel, Chicago.

interaction of the particles with each other, and the ability of the particles to immobilize a film of oil around themselves. Specific surface area is one of the most important factors. Kind and degree of esterification must be considered also.

Elongated particles are more efficient thickeners than spherical ones. Such elongation exists in fibrous or platelike particles or may be achieved by the joining together or reticulation of ultimate spherical units. Particles consisting of networks of such reticulated ultimate spherical units in which the pore-diameter is greater than about 4 millimicrons are also particularly effective thickeners. The smaller the ultimate spherical units forming the network, the more complex the labyrinth obtainable and the more oil which can be immobilized by a given weight of thickener. Thus, it is observed that, other factors being relatively constant, the thickening efficiency is improved markedly as the unit particle size decreases as indicated by increased specific surface area. Thickening efficiency decreases somewhat as longer chain ester groups are used and also as the substrate is more completely esterified.

In general, using estersils having a specific surface area of 25 to 100 $m^2./g.$ esterfied according to the patent, from 20% to 40% by weight of estersil is required to give a medium grease consistency with Mid-Continent solvent treated petroleum oil. In contrast, the same grease consistency is obtained with the same oil using from 8 to 25% by weight of those estersils, similarly esterfied, having substrate specific surface areas of 200 to 400 $m^2./g.$

In the light of the above principles, one will be able to determine by a simple trial or two the amount of a given estersil needed to do the thickening job desired. In general, compositions of the patent are preferably obtained by mixing oil and estersil in an oil: estersil weight ratio in the range of 16:1 to 3:1.

In general, in the mixing and milling operation, the estersil particle size is reduced, as required, to avoid lumpiness. In the case of grease preparation, for example, the estersil structure is broken down and dispersed in the oil to the point where the grease is not lumpy and is unctuous to the touch. The grease compositions begin to feel rather smooth when the gross diameter of the estersil particles is less than about 150 microns. However, since such large particles are merely coherent aggregate of smaller ultimate units, they are usually greatly reduced in size during the milling operation. Thus, in the case of the preferred estersils based on a precipitated amorphous silica substrate having a surface area of at least 200 $m^2./g.$ and a pore diameter greater than 4 millimicrons, milling will reduce the aggregate to units smaller than 5 microns.

The nature of the esterified siliceous component of the oil-estersil compositions of the invention can be determined by first extracting the oil from the composition with a solvent and then analyzing the remaining esterfied siliceous solid to determine its carbon content, identity of the ester grouping, and such properties as surface area, pore size, ultimate particle size, bulk density, and the like.

The term grease, as used in the patent, refers to estersil-oil compositions having a consistency within the range conventionally ascribed to greases heretofore, namely, a micro-penetration value of from 30 to 420 at 77°F. as determined by the method described in ASTM "Standards on Petroleum Products and Lubricants," November 1949, page 1309.

The proportion of hydrogen-bonder employed may also be widely varied depending upon the properities desired in the final product and upon the character of the lubricating oil and estersil used. Generally, the hydrogen-bonding compound is not the preponderant component of the mixture but is, rather, a minor component. The precise proportion of bonder to use in any particular composition can readily be determined by adding various increments and determining the rheological control effected in the final product, the additions being discontinued when the desired properties are reached.

To explain the foregoing observations as to the effect of hydrogen-bonding donor compounds upon the rheological properties of estersil-oil compositions, the patentee has evolved a theory that, upon the siliceous substrates of the esterils, there are "patches" of silanol and/or siloxane groups due either to incomplete esterfication or to partial break-down of the estersil structure occurring when it is milled into the oil, or to both of these factors.

Such unprotected areas of "patches" are capable of interacting, as by hydrogen-bonding, with each other, with components of the mixture, or with moisture from the air. Now, the more of these interactions which occur among the estersil particles, either directly or indirectly through a polyfunctional hydrogen-bonding donor compound such as glycerine, the more complex will be the labyrinth of the solid estersil phase and the more oil will be entrapped therein. This will have the effect of increasing the thickening efficiency of the estersil or increasing the consistency of apparent viscosity of the lubricating composition at a given estersil concentration. The polyfunctional hydrogen-bonder such as glycerine appears to act as a "bridge."

On the other hand, if the patches interact with a hydrogen-bonding donor compound which is monofunctional, or is difunctional but incapable of acting as a bridge, such as monoglyceryl stearate, interactions between estersil particles will be reduced or eliminated, and this will result in a peptization or decrease in the thickening effect of the estersil phase.

The products appear completely homogeneous upon examination with the unaided eye or under a light microscope. No grit is visible. Often, especially when the preferred estersils having an internal structure of amorphous silica are used, the products are clear and transparent, thus, having improved appearance over

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conventional oil-metal soap greases and the usual opaque mixtures of lubricating oil and inorganic fillers.

The grease compositions of the patent have a buttery texture if the oil used in the composition is of moderate or low viscosity. With relatively high viscosity oils, say around 2000° SUS at 100°F., greases having a more tacky mixture are obtained.

The grease-like, and the semi-solid compositions are particularly characterized as being substantially non-melting, even up to temperatures at which the lubricating oil phase will ignite. They also are claimed to possess excellent shear stability. This is illustrated by the fact that there is little or no change in their consistency as determined by the ASTM micropenetration method after working in such devices as the Hain microworker described in Naval Research Laboratory Report TP-2817, April 1946, "A Microworker for Lubricating Greases," by G. M. Hain.

The excellent lubricating properties and lack of grittiness or abrasiveness of the grease products have been demonstrated in wear tests in comparison with conventional soap greases using standard machines such as the Almen, Timken, and Cornell testers.

Example

A siliceous substrate was prepared as follows:

One volume of a solution of 0.48 N sulfuric acid was added at a uniform rate over a period of 30 minutes, at a temperature of about 30°C., to three volumes of a solution of sodium silicate containing 2% SiO_2 and having a molar SiO_2:Na_2O ration of 3.36:1. The sulfuric acid solution was chemically equivalent to 80% of the Na_2O in the sodium silicate solution. Vigorous agitation was provided to insure complete and instantaneous mixing and the temperature of the reacting mass was maintained below 40°C. throughout.

TABLE I

Hydrogen-bonding Donor Compound	*Percent Bonder on Grease*	*Initial Worker Micropenetration at 77° F.*	*Worked Micropenetration and Gel Rating at 77° F. after storage at 210-220° F. for Days Indicated*			*Rust Prevention Test Rating*
			Micropen	*Gel Rating*	*Days*	
Control	None	89	94	Definite Gelation	5	Fair–Good
Water	0.2	78	69	Definite Gelation	10	Excellent
Water	3.0	67	65	Definite Gelation	10	Excellent
I. Monofunctional Alcohols						
Octyl alcohol	3.3	180	91	Slight gelation	4	Fair–Good
Stearyl alcohol	0.17	95	133	Very slight gelation	2	Fair–Good
Stearyl alcohol	1.7	188	Semi-fluid	No gelation	2	Fair–Good
Hydroabietyl alcohol	2.4	107	159	No gelation	12	Fair–Good
II. Polyfunctional Alcohols						
1,3 propanediol	4.1	71	65	Definite gelation	10	Excellent
Glycerine	0.17	76	77	Definite gelation	9	Excellent
Mannitol	3.3	69	62	Definite gelation	4	Good
III. Monofunctional Amines						
Dodecyl amine	3.1	221	155	No gelation	8	Excellent
Octadecyl amine	4.5	Semi-fluid	Semi-fluid			
IV. Diamines						
Decamethylene Diamine	0.17	74	73	Definite gelation	8	Good
Decamethylene Diamine	8.5	40	37	Definite gelation	10	Excellent altho grease structure separated.
V. Ethers						
Di-n-butyl ether	3.6	97	61	Very slight gelation	28	Good
VI. Esters						
Tricresyl phosphate	3.3	98	82	Very slight gelation	8	Good
Tributyl phosphate	1.7	118	122	No gelation	7	Good
VII. Difunctional Carboxylic Acids						
Succinic acid	0.10	77	57	Definite gelation	10	Good

During the acid addition, the pH dropped from 11.3 to about 9 and the sodium ion concentration remained below 0.3N thruout the process. A clear sol resulted.

The sol obtained by the above step contained 1.5% SiO_2. The solids in the sol consisted of discrete particles of silica having an average diameter less than 5 millimicrons, too small to be measured by the electron microscope.

The sol was heated to 95°C. Solutions of sodium silicate and sulfuric acid were added simultaneously at a uniform rate over a period of two hours. The sodium silicate solution added contained 10% SiO_2 and had a molar SiO_2:Na_2O ratio of 3.361. The sulfuric acid was a 4% aqueous solution and was added in amount sufficient to neutralize 80% of the Na_2O in the silicate solution. The addition of silicate and acid was continued until one part of SiO_2 had been added for each part of SiO_2 present in the initial sol. During the addition, vigorous agitation was employed. The pH of the mixture slowly rose from 9 to 10 during the additions and was then maintained at about 10. The sodium ion concentration remained below 0.3 N thruout the process.

During the heating of the initial sol and the subsequent addition of silicate and acid, the tiny discrete dense ultimate articles of silica increased in size; they became chemically bound together in the form of open networks or coherent aggregates of supercolloidal size. This action is called the "build-up" step. The aggregates precipitated so that the resulting mass was in the form of a slurry.

To aid filtration, the slurry was further flocculated with a 2% solution of a mixture of cetyl and lauryl trimethyl ammonium bromide, 0.16% of the mixed compounds being added, based on the weight of the silica. The slurry was filtered and the wet filter cake reslurried in water. The reslurry was adjusted to about pH 7 with dilute sulfuric acid, then filtered, and the filter cake washed with water. The undried filter cake as obtained on a vacuum filter contained about 12.5% SiO_2 by weight. The specific surface area of a sample of this substrate material, after drying in air at 120°C., was approximately 300m². g. as measured by the aforementioned nitrogen adsorption method.

The esterification of the substrate was carried out as follows:

Twenty-two hundred grams of the wet filter cake containing about 275 grams of silica was slurried in 6 liters of n-butanol. The slurry was placed in a 12-liter, three-necked flask equipped with an electric heating mantle, a thermometer, a mechanical stirrer, and a three-quarter inch column three feet long, packed with 1/8 inch glass helices.

The slurry was heated and material allowed to distill at a reflux ratio of about 2:1 until the distillate no longer separated into two layers and the pot temperature had risen to above 116°C. indicating that most of the water had been removed by azeotropic distillation. The heating and distillation required about 13 hours.

The slurry was then transferred to a three-gallon stainless steel autoclave and heated to 200°C. under autogenous pressure. When the temperature was reached the heat was cut off and the autoclave allowed to cool to room temperature. The heating required about 2.5 hours.

The water content of the alcohol phase of the slurry in the autoclave, at the end of the treatment, was about 0.2%. The slurry was filtered and the filter cake dried at 75°C. in a vacuum oven for about 24 hours.

The dried material was a fluffy white powder. It was organophilic and hydrophobic. It had a specific surface area of 293m²./g. It showed no adsorption of methyl red dye. The bulk density of the estersil product was 0.134g./cc. under a compressive load of 3 p.s.i. Analysis showed that the product contained 87.67% SiO_2 and 6.56% carbon. After heating the sample at 500°C. to remove the ester coating, the specific surface area of the substrate was found to be 335 m²./g. From this value it was calculated that the original estersil had 280 butoxy groups per 100 square millimicrons of substrates surface.

A grease was prepared using the estersil obtained above by mixing the estersil with a Mid-Continent solvent-treated petroleum oil (viscosity 300 SUS at 100°F., viscosity index=100) in an oil-estersil weight ration of 7.5—that is, the mixture contained 13.3% of estersil by weight.

The estersil was initially worked into the oil using a mortar and pestle until a fairly homogeneous mixture was obtained. The mix was then passed thru a Kent three-roll ink mill with the rolls set for clearance of 0.0015 inch. Seven passes thru the mill were made to insure complete mixing and give a homogeneous product. The grease obtained was clear and buttery. Its consistency was equivalent to about a No. 2 to No. 3 grade cup grease. It had a micropenetration value of 89 at 77°F. as measured by the ASTM micropenetration method (ASTM Bulletin No. 147, August 1947, pages 81-85).

After five days storage at 210 to 222°F. and subsequent cooling to 77°F. the grease exhibited definite gelation and had a worked micropenetration of 94. A sandblasted mild steel test panel was coated with a 1/16" to 1/8" layer of the above grease, placed in a wooden rack, and exposed at 100% relative humidity 120°F. for 150 hours. Thereupon, inspection showed that the grease had hardened appreciably and some mud-cracking had occurred near the point of support which allowed water to undercut the grease layer and cause corrosion in an area which extended up to 1/2" from the point of support. Since the rest of the panel was completely protected a rating of fair to good was assigned to this grease.

Control of the rheological properties of a grease prepared in an identical manner was effected by adding

a hydrogen-bonding donor compound—namely, water. The water was added in the proportion of 0.2% by weight based on the weight of grease and replaced 0.2% of the mineral oil, the estersil concentration thus remaining constant. After adding the water the grease was ink milled and the micropenetration at 77°F. was ascertained to be 78.

After 10 days storage at 210 to 220°F., the general appearance of the grease was excellent and the worked micropenetration after cooling to 77°F. was 69. The above data, indicate the improvement in thickening efficiency afforded by the addition of the water and the stability of the resultant grease to changes in permanent consistency upon elevated temperature storage.

The control or rheological properties was further demonstrated by the fact that the grease, as a layer, had a protective effect against corrosion of sand-blasted mild steel panels. Thus, a coating of the grease 1/16 to 1/8" thick was placed on a clean, mild steel test panel and the panel was subjected to exposure to 150 hours at 120°F. and 100% relative humidity, as previously described. After exposure it was found that there was no corrosion of the test panel within 1/8 inch from the edge of the panel or from the point of support, apparently because the hardening and mud-cracking phenomena had been minimized through proper control of rheological properties. Since it is conventional practice in this corrosion test to ignore corrosion within 1/8" of the edge of the test panel because of discrepancies in coating, rust prevention was considered excellent.

The various types of rheological control obtained by the addition of the hydrogen-bonding donor compounds will be seen from Table 1. Thus, water and the polyfunctional additives increased the thickening efficiency of the estersil and therefore tended to pre-harden the grease with a resultant improvement in rust prevention properties. The monofunctional additives such as the monoamines and monoalcohols tended to reduce the thickening efficiency of the estersil and thereby demonstrated their probable effectiveness for preparing stable dispersions of the estersils at low concentrations in lubricating oils or hydraulic fluids. The relatively weak hydrogen-bonding agents such as the ethers and esters, reduced grease gelation at elevated temperature and thus improved lubricating properties in the types of anti-friction bearings where feeding characteristics are of prime importance.

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Saponification No.	120-130
Free Inorganic Acid	0.2% max.
Iodine Value	20-40
Apparent Solidification Point (titre)	Approx. 44° C.
Softening Point	45-48° C.
% Sulfur	No corrosive sulfur

A.O.C.S. Methods

PEOPLE in the Industry

Southwest Grease Elects Simmons Vice-President

At the annual meeting of the board of directors of the Southwest Grease & Oil Co., Inc., held in Wichita, Kansas, Wilson Simmons, sales manager, was elected to the newly created office of vice-president, sales.

An approximate 30 per cent increase in dollar sales for the year-to-date reported at the meeting partially set the stage for this promotion.

With the company's new plant No. 2 scheduled to start production by January 1, 1957, a tremendous sales job is facing the House of "GOOD" Grease, and this new position was created to place added importance upon it. In an interview shortly after his election, Simmons confidently predicted further increase in the sale of company products and established a 1957 goal 10 per cent above this year's figure.

Beman and Richter Head Socony Mobil Chemicals Department

Warren A. Beman has been appointed to head the marketing division and Frederick P. Richter has been named head of the technical division of the recently established petroleum chemicals department of Socony Mobil Oil Company, Inc., Park W. Judah, manager of the department, has announced.

Mr. Beman has been head of the process products group in the industrial division of Socony Mobil's lubricating department since 1954. A chemical engineering graduate of Purdue University, he joined the company in 1942.

Dr. Richter formerly was supervisor of the chemical section of the company's research and development laboratory at Paulsboro, N. J. More recently he has been a member of the technical staff of the manufacturing department of General Petroleum corporation, Socony Mobil's West Coast affiliate. He holds A.B. and M.A. degrees in chemistry from Temple University, and received his Ph.D. in chemistry from Illinois University.

Bogdanoff Will Represent Swift

B. John Bogdanoff has been appointed New York state field representative for the industrial oil department of Swift & Company, Hammond, Indiana.

Bogdanoff's responsibilities will include the sale of Swift's complete line of fatty acid and industrial oils throughout New York State with the exception of New York City, Fred Beneker, general sales manager, announced.

Bogdanoff is a graduate of the University of Wisconsin and he served in Swift's research laboratories before joining the sales department. He will make his residence in Syracuse.

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Socony Mobil Appoints McDaniel

The position of assistant for dealer-distribution relations to the director of domestic marketing has been created by Socony Mobil Oil Company, Inc. George D. McDaniel, general manager of the company's eastern region for the past ten years, has been named to the new job, Vernon A. Bellman, director of domestic marketing, has announced.

Mr. Bellman also announced the appointment of Glenn L. Werly as domestic marketing manager, the post Mr. Bellman held until he was elected to the company's board of directors on November 26. Mr. Werly has been manager of the retail merchandising department for ten years.

Other appointments made by Mr. Bellman:

Athur W. Warner to succeed Mr. McDaniel as eastern region manager. Mr. Warner has been manager of the Philadelphia division for eight years.

Quentin W. Regenstein to a new position of administrative assistant to the domestic marketing manager. Mr. Regenstein has been manager of the New York City division for twelve years.

Harold E. Jenkins to succeed Mr. Werly as manager of the retail merchandising department. He has been merchandising manager in the department for seven years.

Battelle Names Allen Engineering Consultant

C. Malcolm Allen has been appointed consultant in the engineering mechanics division of Battelle Institute, Columbus, Ohio.

Allen, who is a specialist on bearing problems and the related fields of friction, lubrication, and wear phenomena, will direct his attention to the technical planning of Battelle's research programs.

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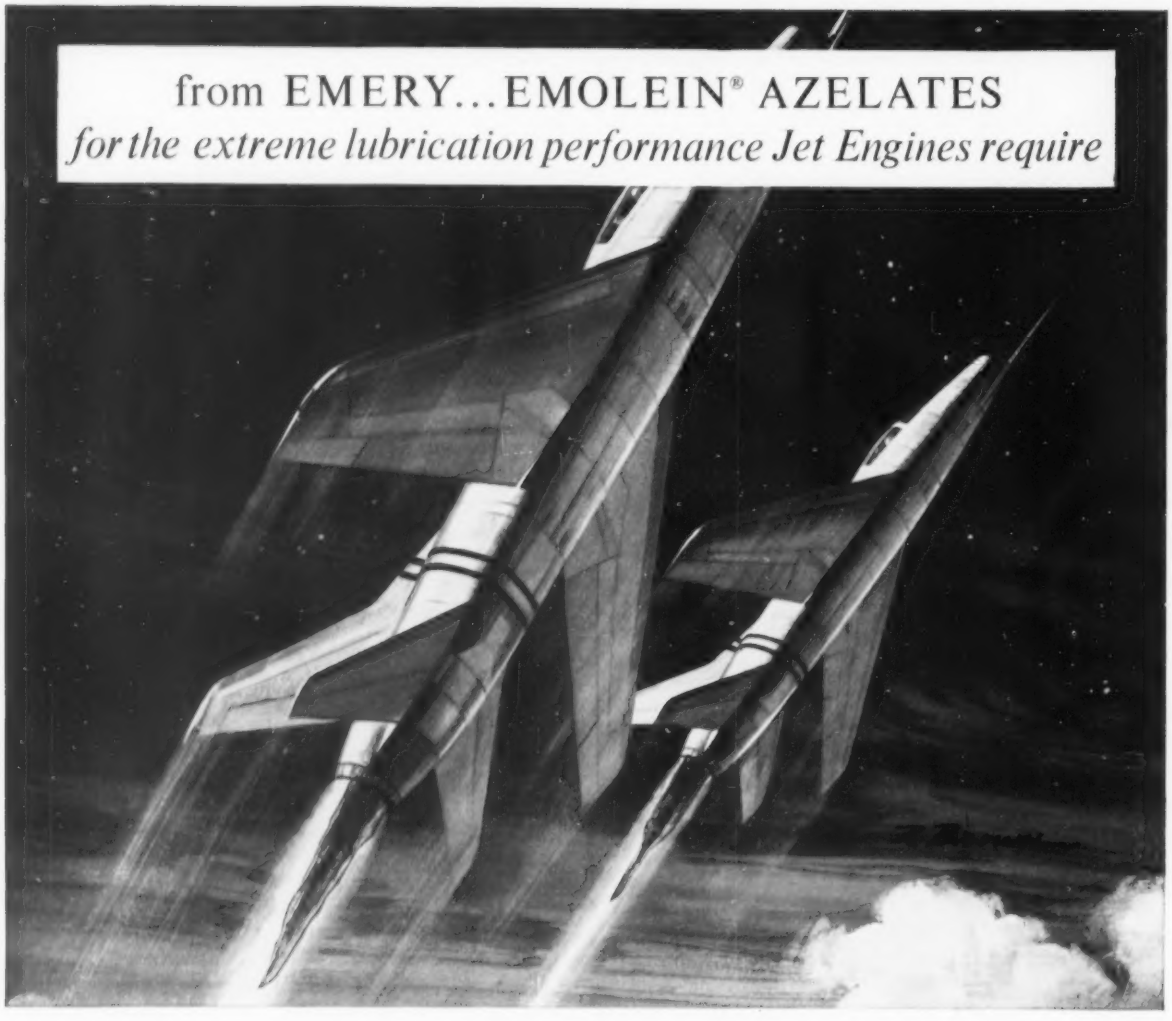

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Since the Emolein azelates are based on azelaic acid obtained from domestic fats and oils abundant in supply, availability is not contingent on strategic imported raw materials. Also, future plant expansion is being carefully coordinated with the broadening utility of these diesters.

Application in other synthetic lubricants

Other military specifications met by synthetic fluids and greases compounded from Emolein Azelates include: Mil-G-3278 A, Grease, Aircraft and Instruments-Low and High Temperatures; Mil-L-6085 A, Lubricating Oil, Aircraft Instruments, Low Volatility; and Mil-L-6387 A, Lubricating Oil, Synthetic Base.

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F. J. Snyder, Fiske President, Deceased

Frederick J. Snyder, Sr., president of the Fiske Brothers Refining company of Newark, New Jersey, and Toledo, Ohio, died at his home in Spring Lake on November 25. He spent his entire career in the petroleum industry and in 1953 celebrated his 50th anniversary with Fiske Brothers. He was a member of the New York Oil Trade Association and was one of its organizers, serving as the third president in 1920 and 1921.

Fiske Brothers was one of the first members of the National Lubricating Grease Institute and shortly after joining the institute Snyder was voted treasurer in 1938.

Continental Can and Gair Announce New Officers

General Lucius D. Clay, chairman of the board of Continental Can company, has announced that George E. Dyke, formerly chairman of the board and president of Robert Gair company, Inc., which merged October 26 with Continental Can company, Inc., has been elected vice chairman of the board of Continental.

Three new vice presidents of Continental Can were also elected at the board of directors meeting: Raymond F. DeVoe and Norman F. Greenway, former senior vice presidents of Gair; and Robert L. Fitts, a Gair vice president.

Robert Gair company, a foremost producer of paperboard and paper packaging products, was merged into Continental after overwhelming votes of approval by all stockholders concerned. It is now operated under the Continental name.

General Clay announced that the former activities of Robert Gair, together with Continental Can's paperboard making and fibre drum divisions, will be administered under the general supervision of Mr. Dyke as vice chairman of the board. The newly named vice presidents will exercise their responsibilities within these general areas.

Campbell Joins Kerr-McGee

Odell Campbell, former manager of laboratories for E. I. du Pont de Nemours and Company in Tulsa, has joined Kerr-McGee Oil Industries, Inc., Oklahoma City, as technical assistant to R. M. Chesney, vice president in charge of refining.

American Lithium Institute Announced

Establishment of the American Lithium Institute, Inc., for the purpose of conducting research on lithium and its compounds, it has been announced by American Potash and Chemical Corporation, Foote Mineral Company and Lithium Corporation of America. In a joint statement by the three major

Sittig Elected Vice President

U. S. Lithium producers, the companies stated that the Institute has been formed to meet the growing necessity for a central organization staffed to direct research on problems of an industry-wide nature. The Institute will also act as an agency for the correlation and dissemination of technical information on lithium.

Institute Offices in New Jersey

The Institute—a non-profit organization—will maintain offices in Princeton, N. J. A technical library and literature service will be maintained at this office.

Marshall Sittig, formerly of Ethyl Corporation, has been elected president and managing director of the Institute. Mr. Sittig is widely known for his many technical writings in the petroleum field. He is an authority on the alkali metals.

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Emery 3033-S is a new-type dipropylene glycol diester based on pelargonic acid, a unique, C_9, saturated, monobasic acid.

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Since 3033-S is based on a relatively low-cost acid, pelargonic acid, long-range economics are favorable. Also, availability is not contingent on strategic, imported raw materials since pelargonic acid is made from abundantly available domestic fats and oils.

Though 3033-S is in a development stage, it is available in tankcar quantities on reasonable notice.

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Cope Directs Van De Graaff Accelerator

A two-million-volt Van de Graaff particle accelerator for exploratory research in petroleum has just been installed at the Richmond laboratories of California Research corporation, a subsidiary of Standard Oil company of California.

The machine, purchased from High Voltage Engineering corporation, Cambridge, Mass., will be used in programs dealing with the effects of radiation on petroleum and its products and on acceleration of reactions leading to useful products from petroleum, according to A. H. Batchelder, vice president, Richmond laboratories.

Work is under direction of Dr. J. Q. Cope, assistant general manager and manager-chemicals research, development and technical service at the Richmond facility.

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Battenfeld Appoints Krause Vice-President

Battenfeld Grease & Oil corporation announce the appointment of George W. Krause to the position of vice-president, in charge of research and production.

Mr. Krause is a native of Oshkosh, Wisconsin and attended Lawrence college, Appleton, Wisconsin. Upon completion of his formal education, he was employed as a chemist by the Standard Oil company of Indiana. He progressed through numerous positions, among which was twelve years as lubrication engineer, contacting heavy industry, to that of the company's assistant manager of the lubricating and industrial sales department. Mr. Krause held this position from 1948 to 1953, when he became president and remained with this firm until his present association with Battenfeld.

The Krause family consists of Mrs. Krause, a daughter, Janice, who is a junior at Lawrence College and a son who is a Lieutenant J.G., US.N.R., stationed at Patuxent River, Maryland.

Mr. Krause comes to Battenfeld, fully experienced in all phases of the lubricating grease industry.

Austin Retires from U. S. Steel Vice Presidency

David F. Austin, executive vice-president—commercial, United States Steel corporation, retired December 31 after nearly 39 years of service, it was announced by Clifford F. Hood, president of the corporation. At the same time, Mr. Hood announced that Mr. Austin's successor will be Richard F. Sentner, presently assistant executive vice president—commercial.

Mr. Austin began his career with United States Steel in 1918 in the New York office of the corporation. After serving in minor capacities, he held several sales assignments, served as manager in three district sales offices, and in 1938 was elected vice president in charge of sales of the Carnegie-Illinois Steel corporation.

During World War II, Mr. Austin joined the War Production Board and served successively as executive consultant, assistant director, deputy director, and acting director of the iron and steel division.

Returning to the vice presidency of Carnegie-Illinois Steel corporation in July, 1943, he was elected vice president of the United States Steel corporation of Delaware in 1945. In January, 1951, Mr. Austin became executive vice president—commercial of United States Steel company and since January, 1953, has held the same position with United States Steel corporation.

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American Potash Appoints Holt

Benjamin M. Holt has been appointed to the newly-created post of project director of the planning and development department of American Potash & Chemical Corporation, according to an announcement by Daniel S. Dinsmoor, AP&CC vice president in charge of planning and development.

In his new position, Holt will be in charge of special major projects being developed by the company, operating under Dinsmoor's direction.

Holt formerly was with the Ralph M. Parsons company prior to his recent joining of American Potash & Chemical corporation.

He was graduated from Stanford University, at Palo Alto, Calif., with a B.A. degree, later receiving an M.S. degree from Massachusetts Institute of Technology, at Cambridge, Mass.

HARSHAW LEAD BASE

Harshaw Lead Base, as an additive to petroleum lubricants, improves extreme pressure characteristics and imparts the following desirable properties:

- Increased film strength
- Increased lubricity
- Improved wetting of metal surfaces
- A strong bond between lubricant and metal surfaces
- Resistance to welding of metals at high temperatures
- Moisture resistance and inhibits corrosion

Harshaw Lead Bases are offered in three concentrations to suit your particular needs:

Liquid 30% Pb	Liquid 33% Pb	Solid 36% Pb

Other metallic soaps made to your specifications. Our Technical Staffs are available to help you adapt these products to your specific needs.

THE HARSHAW CHEMICAL CO.
1945 E. 97th Street • Cleveland 6, Ohio
Branches in Principal Cities

Eastern Sales Realignment

A realignment of the eastern sales department of American Potash & Chemical corporation into two operating groups has been announced by William J. F. Francis, vice-president in charge of sales.

Under the new arrangement the department will operate as a heavy chemicals division and an industrial chemicals division. Ed M. Kolb has been named as general sales manager of the heavy chemicals division, while Dr. Al J. Dirksen, formerly director of AP&CC's market development department, has been appointed general sales manager of the industrial chemicals division. Headquarters will remain at the company's eastern general sales office in New York.

Clines Named Sales Manager

William M. Clines, formerly western sales manager, was named general sales manager, western, to handle all company sales in the western area. Clines will continue to headquarter at AP&CC's executive offices at Los Angeles.

Francis said the changes were made because of American Potash & Chemical corporation's steadily expanding sales activity throughout the United States.

Emery Appoints Killheffer To Research Staff

The appointment of John V. Killheffer, Jr., to the research staff of Emery Industries, Inc., Cincinnati, is announced by Dr. R. G. Kadesch, director of research. He will be associated with the plasticizer group of the process research section under the supervision of Dr. C. A. Sprang, where he will work on both new and current plasticizers of Emery's extensive line.

Prior to joining Emery, Mr. Killheffer was a chemist with the organic chemicals department of E. I. du Pont de Nemours & Company. He holds the degrees of B.S. from the Massachusetts Institute of Technology and M.S. from the University of North Carolina, and is a member of the American Chemical Society and Alpha Chi Sigma.

Du Pont Appoints Miller

Charles Miller has been appointed regional laboratory manager in Tulsa, Oklahoma. Miller holds a Masters Degree in chemistry from the University of Delaware. He has served as technical assistant in the mid-continent regional laboratory for the past year. He joined Du Pont in 1941 and was first assigned to Jackson laboratory, one of the foremost organic chemical laboratories in the country. He was associated with Du Pont's petroleum chemicals division in its petroleum laboratory for several years prior to 1955 when he was assigned to the Tulsa laboratory.

Through Miller the expanding technical services of the petroleum chemicals laboratory will continue to be made available to the industry.

News continued on page 51

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CORRECT LUBRICATION

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Makers and Marketers of
Mobil Automotive
Products
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Socony Mobil Oil Co., Inc.
and Affiliates: MAGNOLIA PETROLEUM CO.
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you can depend on
PENOLA AUTOMOTIVE LUBRICANTS

for top quality performance
in every lubricating job.

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Industry NEWS

Kerr-McGee Constructs New Company Headquarters

Construction is scheduled to start soon on a $1.9 million office building expansion for Kerr-McGee Oil industries, Inc., Oklahoma City.

The company's headquarters building will be enlarged from approximately 56,000 square feet of floor space to 173,000 square feet, in a project that calls for remodeling of two buildings and construction of an eleven-story tower connecting them.

The present Kerr-McGee building is an eight-story structure on the northeast corner of NW Second and Robinson streets. Last spring the company purchased the old Oklahoma City YMCA building in the same block, and a two-story building intervening.

The two-story building will be torn down and the eleven-story aluminum-and-glass tower built in its place. The two buildings thus joined will also be sheathed in aluminum, blending the structures into a single modernistic building.

A helicopter landing deck will be built on one section of the roof. The swimming pool and basketball court of the former YMCA building will be used as a part of the Kerr-McGee employee recreation program. Completion is scheduled for the end of 1957.

National Petroleum Bibliography Releases Index

The first edition of a completely new type of periodical publication devoted to the petroleum industry is off the press, according to an announcement from National Petroleum bibliography, publisher.

Designed for use as a desk top library file by executives, petroleum technologists and researchers, the National Petroleum bibliography is a comprehensive bi-monthly index of literature and data pertinent to all oil industry operations.

More than 400 publications per issue, predominantly technical and trade journals, are being indexed in the new bibliography, which also includes current books, technical papers, maps and various special publications of oil and gas agencies and institutions.

Bibliographical listings are indexed under the Uren decimal system, widely used by oil companies in filing technical data. Information relative to all divisions of the industry is classified under under specific numbers and headings.

A sample copy of the 121-page edition may be obtained by writing National Petroleum bibliography, Box 3586, Amarillo, Texas.

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Consistently uniform in metallic content and viscosity

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Non-Oxidizing - - - contains no unsaturated soaps

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API Releases Study of Research and Predictions In Oil Industry

New York—A new study which emphasizes not only the rapid growth but also the extensiveness of research and development in the oil industry has been published by the American Petroleum Institute.

Among the highlights of the detailed study are these points:

. . . the oil industry, in 1955, spent more than $160 million dollars on research and development work;

. . . of this, development of new products and/or processes accounted for 52 cents out of each dollar; improvements of existing products and/or processes, 37 cents; and fundamental research, 11 cents;

. . . oil industry research currently is utilizing the time and talents of almost 15,000 persons;

. . . of these, more than one-third are scientists or engineers, and another one-third are trained technicians;

. . . of the scientists and engineers, more than one-half are chemists and chemical engineers.

The API study has been published as "Oil Industry Background Information Bulletin No. 14—Research and Development Expenditures and Personnel in the Oil Industry."

DuPont Devises "Billion Dollar Briefcase"

The "Billion Dollar Briefcase," a new lecture demonstration of the interdependence of large and small business, has been developed as a service to the oil industry by the Du Pont Company's petroleum chemicals division.

Using illustrations largely from petrochemical products, "Briefcase" explains the role of big business in the American economy. It brings out that size is related to function, showing how large companies build opportunities for small companies by undertaking the jobs the little one cannot do, while big business is dependent on the essential operations which small firms are best qualified to perform.

The "Billion Dollar Briefcase" was created as a supplement to the industry's now-famous "Magic Barrel." Like the latter, it was developed by H. C. Thompson, assistant sales promotion manager of Du Pont's petroleum chemicals division. The new presentation, which was reviewed in September by the national Oil Industry Information Committee, promotes better public understanding of America's free enterprise system.

In the preface to the "Billion Dollar Briefcase" script, to be provided to some 1,200 "Magic Barrel" speakers throughout the nation, it is pointed out that even though our present business system has enabled more people to gain more things than any other yet devised, its existence, and that of the oil industry as a cornerstone of this system, is continually threatened and unfairly challenged.

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HYDROGENATED FATTY ACIDS

TALLOW FATTY ACIDS

VEGETABLE FATTY ACIDS

HYDROGENATED GLYCERIDES

HYDROGENATED CASTOR OIL

12 HYDROXY STEARIC ACID

H-14

HARCHEM DIVISION

WALLACE & TIERNAN INC.

SUCCESSOR TO W. C. HARDESTY CO., INC.

25 MAIN STREET BELLEVILLE 9, NEW JERSEY

Cities Service Exhibits at Chicagoland Fair

Cities Service Oil company will be one of the leading exhibitors at the Chicagoland fair, gigantic business, industrial, and cultural exposition to be held at Navy Pier, Chicago, next June 28-July 14.

Sponsored by the Association of Commerce and Industry, the Chicagoland fair will feature five miles of colorful exhibits and pageants extolling Chicago as a "land of opportunity" and saluting the city's progress. Hundreds of companies are expected to display their products and services at the exposition.

An estimated 500,000 persons are expected to attend the seventeen-day fair. They will view the many colorful exhibits, see a special pageant, and enjoy other entertainment features.

Lincoln Announces New Industry Aids

Grease Gun for Disposable Lube Cartridges

An all steel, high pressure lever gun by Lincoln Engineering is now available for use with the handy, new 14¼ oz. grease cartridges. Cartridges provide easier and faster reloading without waste and the lubricant stays refinery pure, according to Lincoln. The gun is equipped with a filler nipple for 30-second refilling by a filler pump or by the conventional suction method. The capacity is 18 oz. using bulk lubricant, and may be spring or force primed. It develops 10,000 p.s.i. maximum pressure. Complete details are given in bulletin 726.

Low Cost, Large Capacity Suction Gun

Lincoln has readied a suction gun designed for high performance at low cost for servicing gear cases, transmissions, differentials or transferring any fluid lubricant. A flexible linpak follower is precision fitted to the barrel and is impervious to lubricants. All steel construction with rust resistant, gun metal blue finish provides maximum durability. Bulletin 223 gives details.

Hi Pressure Lubricator for Containers

An air motor operated lubricant pump, incorporating a steel cover, which can be clamped in 30 seconds to any standard 25 to 50 lb. original refinery container is now ready for use. Lincoln states that the pump will develop grease pressure 50 times greater than air line pressure. It is equipped with a 6 foot pressure hose and control valve with curved extension for contacting hard to reach fittings. Details are described in bulletin No. 727.

Portable Gear Lube Dispenser

A portable, manually operated gear lubricant dispenser, incorporating a steel cover which can be clamped in 30 seconds to any standard 25 lb. to 50 lb. original refinery container has been designed. It includes a 5 foot hose assembly with curved non drip nozzle for easy application. The lubricant output is one pint per 10 strokes and the maximum pressure developed is 400 lbs. For complete details write for bulletin 727. Address requests for all bulletins to Lincoln Engineering Company, 5702-30 Natural Bridge Avenue, St. Louis 20, Missouri.

CONSTANT

SEARCH for BETTER LUBRICANTS by DENCO

ASSURES QUALITY IN YOUR PACKAGE

The same "Know-How" that makes Denco the first in the manufacture of bentone lubricants, is used in everyday production of Denco's complete line of greases, oils, and compounds for every lubricating and metal-working function. A "Know-How" that's backed by fifty years of experience.

We will produce to your specifications, package, label, and ship according to your instructions.

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SWIFT'S INDUSTRIAL OILS INVITE COMPARISON

Write for a trial order of any of these Swift quality products . . . a trial in your own shop will convince you of their stability and dependability in helping to produce lighter, more uniform lubricants.

RED OILS • STEARIC ACIDS • LARD OILS • SPERM OILS • TALLOWS HYDROGENATED CASTOR OILS, GLYCERIDES, & FATTY ACIDS METHYL #12—HYDROXYSTEARATE • #12—HYDROXYSTEARIC ACID VEGETABLE FATTY ACIDS • TALLOW FATTY ACIDS • ACIDLESS TALLOWS

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INDUSTRIAL OIL DEPT.

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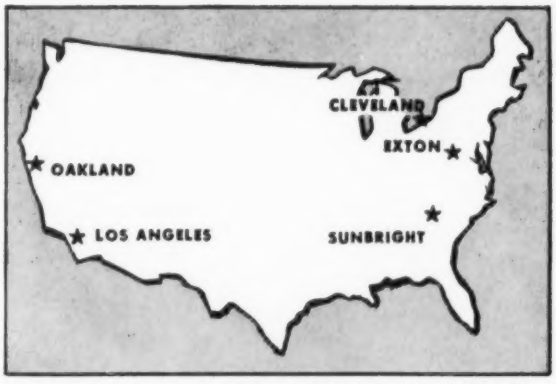
Hammond, Indiana

Foote Lithium Hydroxide Now Available from Five Key Distribution Centers

To provide even better service for grease manufacturers, Foote Mineral Company is now operating five key distribution centers for lithium hydroxide monohydrate, used in the production of multipurpose greases.

Located at Oakland and Los Angeles, Calif.; Cleveland, Ohio; Sunbright, Va.; and Exton, Pa.; these centers will maintain ample stocks of Foote lithium hydroxide to assure the most rapid and advantageous deliveries to all grease manufacturers.

A company spokesman stated that 30 major grease manufacturers are producing lithium base greases that provide positive lubrication at temperatures ranging from −60°F. to over 300°F. even in the presence of water. Other producers are invited to inquire about the distinct manufacturing, marketing and lubricating advantages of these superior lithium multipurpose greases.



Map shows the location of the five distribution centers established by Foote to provide localized supply of lithium hydroxide used as the base in the manufacture of multipurpose greases.

FOOTE MINERAL COMPANY
402 Eighteen W. Chelten Building, Philadelphia 44, Pa.
RESEARCH LABORATORIES: Berwyn, Pa.
PLANTS: Exton, Pa.; Kings Mountain, N.C.; Sunbright, Va.; Knoxville, Tenn.

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tion has performed that function well . . . The Republican Party is not a fit instrument to govern a Nation that wants to make progress for the benefit of the ordinary people."

VISITS SIX COUNTIES

Stevenson earlier had a word about Mr. Eisenhower's personal role in governing the Nation. At

setts for three days. Next he heads west to Washington and California.

The Democratic nominee told his Jersey City audience that President Eisenhower must personally take the blame for "the shortcomings in the public housing program."

He said Mr. Eisenhower's reac-

Stevenson said.

Specifically he accused the Republicans of using their "liberation" policy vis-a-vis European satellite countries for political purposes.

At the eight cities the Democratic Presidential nominee visited, he welcomed the local and Con-

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All the rest of it is in the mind of the lucky ones who see the picture, which is just another way of saying that originally the thought and the whole idea were in the mind of the photographer. Accidents can and do happen, but if your picture expresses something worthwhile and makes the observer think as he sees it, the chances are many millions to one that the photographer had an idea and

THE FALL COLOR MOVIE

Along about this time of year everybody who has a movie camera —and who has not?—goes out to make a movie of the wonderful artistry with which Jack Frost has painted the leaves.

Most unhappily, too many of those movies might better have been made with a still camera. Because of the techniques used, or perhaps the lack of them, the pictures made with the movie camera

even better if father or some other member of the family knows enough about the trees to discuss their differences in closeups that might show leaves or bark or anything else that seems to him, as a naturalist, to be important.

POINT OUT TREES

If the approach is to be entirely scenic, remember that scenes need life in them if there are going to be very many of them. Let somebody walk into the scene to show

Stevenson smiled and said "Speaking of Ike, I'd trust him

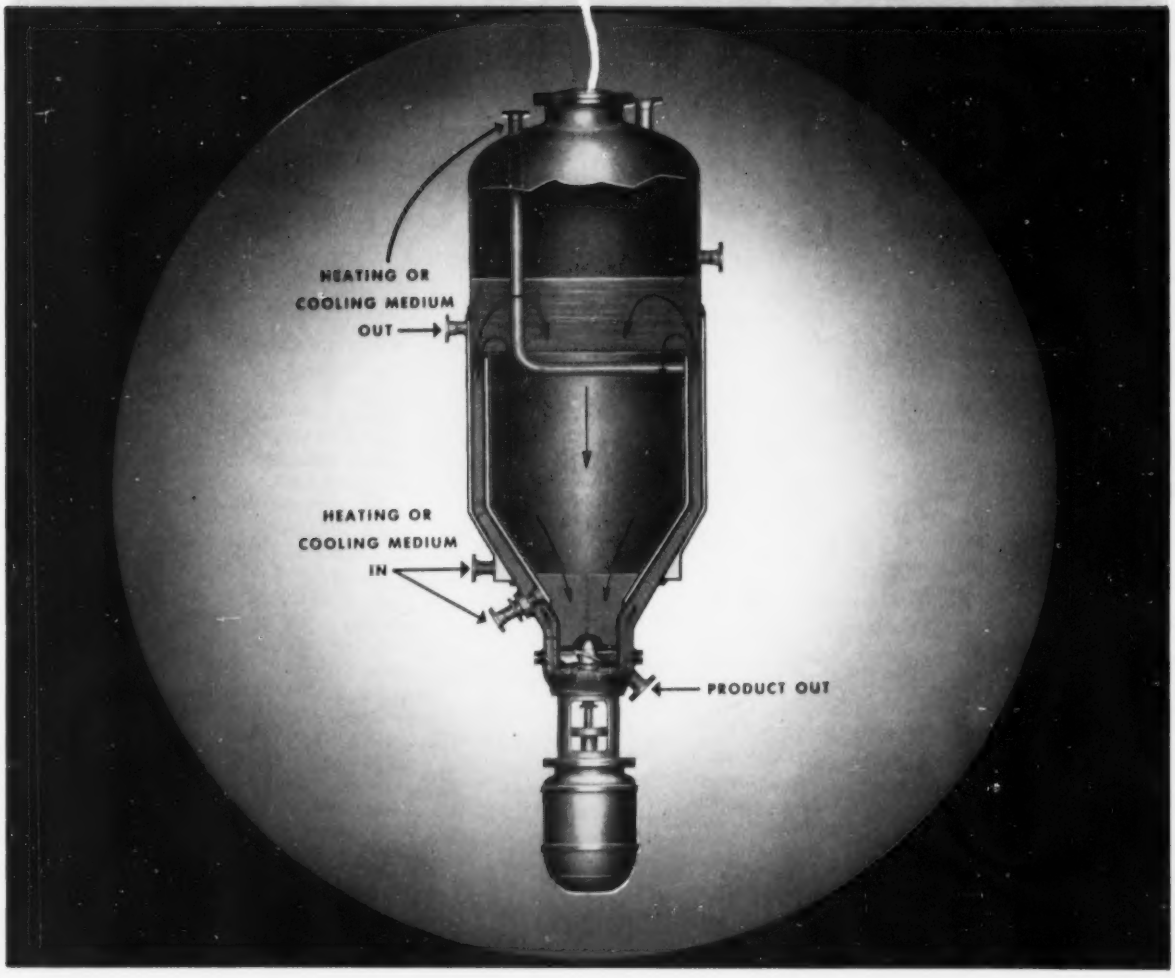
ON SCHOOL NEEDS

When it came to schools, Stev-

Ex-Dancer Dies in Leap

SAN FRANCISCO, Oct. 2 (AP). —A former dancer, apparently

The STRATCO contactor



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Adaptable to existing plants to improve quality of product and reduce manufacturing cost. A vital part of all complete grease plant installations.

Used for complete saponification of all soaps with very short time cycles, resulting in less soap requirement, simplified laboratory control and reduced man hours of operation. Also employed advantageously for sulphurization of oils. Available in capacities from 2 gallon laboratory units to 2300 gallon commercial sizes.

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